CHEMICAL ANALYSIS
FOR
GLASSMAKERS

This Volume

IS AFFECTIONATELY DEDICATED

TO MY MOTHER.

PREFACE.

THE manufacture of glass being essentially an industry requiring chemical operation, it is apparent that its successful prosecution requires some knowledge of chemistry. This fact, however, has not been given the attention it deserves. The skill of the glassmaker heretofore has been dependent on his knowledge of formulæ and recipes, and on his experience with them which enabled him to forecast results without actually knowing the reasons for these results. When unfavorable they were attributed to "bad luck," while as a matter of fact, bad luck was probably due to imperfections in the ingredients used in the batch, which with a little chemical knowledge could have been detected in time to avoid failure in the melt.

Most glassmakers are at the mercy of those who supply the chemicals, and although competition has considerably raised the standards of purity there is room for improvement.

Besides the testing of raw materials there are operations which should be subjected to chemical scrutiny, such as batch-mixing, gas-making, etc. This is best performed by constant, routine analysis, which will be found very valuable in the long run, besides exerting a salutary effect on the workmen.

This book is written for those who have not had the advantage of a training in chemistry, but who while engaged in glassmaking have felt the necessity of scientific supervision of their work.

It is a difficult matter to instruct in chemical analysis one who has had no previous chemical knowledge, and although this book is written in as simple language as possible, it is desirable that the reader, if not a chemist, should obtain practical instruction from some trained operator. This applies particularly to Part III, General Procedure of Analysis, in which the manipulations are difficult to describe with justice, but are quite simple when actually performed.

Most of the methods of analysis given are from the author's actual experience, the others are those commonly used by chemists. I am indebted to Dr. Arthur H. Elliott for many suggestions, for several of the methods, and for much kind assistance.

CONTENTS.

PART I.

CHEMICAL THEORY.

In order to properly understand the various operations of Chemical Analysis, a short description of Chemical Theory will be necessary.

The smallest sub-division into which a body can be divided without its undergoing a chemical change, is called a *molecule.* The smallest particle of a body which can exist in chemical combination is called an *atom.*

Any substance which cannot be divided into other substances having different chemical characteristics is called an *elementary substance* or *element.* A substance which can be so divided is called a *compound.* The atoms which compose an element have the same nature, while those which make up a compound may be of two or more different natures. As an example, we have the metal Iron. No matter how finely we divide a mass of Iron, the sub-division will still be Iron, and have all the properties of that metal. On the other hand, we have common iron rust, which is a combination of Iron with Oxygen, or an Oxide of Iron. We can by chemical means separate the Iron from the Oxygen, thus obtaining two substances, one of which is a metal, and the other a gas.

Atoms combine with each other to form molecules. These combinations take place according to certain rules and in definite proportions. For example, the gas Oxygen combines with the gas Hydrogen to form Water. These gases combine in the proportion of one atom of Oxygen to two atoms of Hydrogen, thus forming one molecule of water.

If we have a compound, and wish to determine the proportion of a certain constituent, we proceed to separate that constituent from the compound, that is, we cause it to assume some form by which it may be isolated, and measured or weighed. Take, for example, a mixture of water and Hydrochloric Acid in which we wish to

ascertain the amount of Chlorine. We add to the mixture a solution of Silver Nitrate in water. The Chlorine will combine with the Silver, forming Silver Chloride, which will separate and may be weighed, and from its weight the Chlorine may be calculated.

In Chemistry the elements are designated by letters, generally the initials of their names. These letters are called *symbols*, and by means of these symbols we are able to write and explain in a graphic manner, the various chemical combinations of these elements. The elements combine in the proportions of their atomic weights, that is, their weights as compared with that of Hydrogen gas taken as 1. For instance, the atomic weight of Oxygen is 16, or the weight of an atom of Oxygen is sixteen times that of an atom of Hydrogen. As two atoms of Hydrogen combine with one atom of Oxygen to form one molecule of water, we write:

$$H_2 + O = H_2O$$

two Hydrogen atoms plus one Oxygen atom form one molecule of water.

When one atom of an element combines with one atom of Hydrogen or its equivalent, that element is known as *Monovalent.* When one atom of an element combines with two atoms of a monovalent element it is called *Divalent;* with three atoms, *Trivalent,* etc. The following will explain:

HCl, here Chlorine (Cl) is monovalent.

H_2O, here Oxygen (O) is divalent.

NH_3, here Nitrogen (N) is trivalent.

This property of elements is termed *Valence* or Quantivalence, and represents their combining powers in relation to that of Hydrogen.

Elements may possess different valences; for example: Chlorine (Cl) in Hydrochloric Acid (HCl) is monovalent, while in Potassium Chlorate ($KClO_3$) it is pentavalent. Sulphur (S) in Hydrogen Sulphide (H_2S) is divalent, while in Sulphur dioxide (SO_2) it is tetravalent, and in Sulphur trioxide (SO_3) it is hexavalent.

PART II.

THE METRIC SYSTEM.

THE weights and measures given in this book are of the Metric System. This system is used in all operations of chemical analysis. Being entirely of decimal notation it is the most convenient that could be devised, and is coming into universal use even for the common purposes of life. It is the standard system of weights and measures in all civilized countries except the United States and the British possessions.

Its fundamental unit is the *Meter*, which is the distance between two lines, at 0 degrees Centigrade, of a certain bar of platinum-iridium alloy kept at the International Bureau of Weights and Measures, near Paris, France, and is known as the International Meter. The meter is divided into ten equal parts, called *decimeters* (dm.), each decimeter into ten equal parts called *centimeters* (cm.), each centimeter into ten equal parts called *millimeters* (mm.). These divisions are expressed thus: 1 meter equals 10 decimeters or 100 centimeters, or 1000 millimeters. Written decimally, 1 millimeter (1 mm.) equals .001 meter; 1 centimeter (1 cm.) equals .01 meter; 1 decimeter (1 dm.) equals .1 meter.

The metric unit of volume or capacity is the Liter, which is the volume of the cube of one-tenth of the international meter. The liter is divided into ten equal parts called *deciliters* (dl.); the deciliter into ten equal parts called *centiliters* (cl.); the centiliter into ten equal parts called *milliters* (ml.), or *cubic centimeters* (c.c.). These divisions are expressed thus: 1 Liter equals 10 deciliters, or 100 centiliters, or 1000 cubic centimeters. Written decimally, 1 cubic centimeter (1 c.c.) equals .001 liter; 1 centiliter (1 cl.) equals .01 liter; 1 deciliter (1 dl.) equals .1 liter.

The unit of weight is the *Gram* which is the weight of $\frac{1}{1000}$ part of a certain cylinder of platinum-iridium alloy kept at the International Bureau of Weights and Measures, near Paris, France, and

known as the International Kilogram. The gram is divided into ten equal parts called *decigrams* (dg.); the decigram into ten equal parts called *centigrams* (cg.); the centigram into ten equal parts called *milligrams* (mg.). These divisions are expressed thus: 1 gram equals 10 decigrams, or 100 centigrams, or 1000 milligrams. Written decimally: 1 milligram (1 mg.) equals .001 gram; 1 centigram (1 cg.) equals .01 gram; 1 decigram (1 dg.) equals .1 gram.

The terms decimeter, deciliter, and decigram, are not generally employed. Units of length are expressed in meters, centimeters, and millimeters, units of volume in liters and cubic centimeters, units of weight in grams and milligrams.

The following table gives a complete view of the Metric System, showing also the measures which are in excess of the Meter, Liter, and Gram:

Meters.	Names.	Symbols.	Liters.	Names.	Symbols.	Grams.	Names.	Symbols.
10000	Myriameter	Mm.	10000	Myrialiter	Ml.	10000	Myriagram	Mg.
1000	Kilometer	Km.	1000	Kiloliter	Kl.	1000	Kilogram	Kg.
100	Hectometer	Hm.	100	Hectoliter	Hl.	100	Hectogram	Hg.
10	Dekameter	Dm.	10	Dekaliter	Dl.	10	Dekagram	Dg.
1	Meter	M.	1	Liter	L.	1	Gram	G.
.1	decimeter	dm.	.1	deciliter	dl.	.1	decigram	dg.
.01	centimeter	cm.	.01	centiliter	cl.	.01	centigram	cg.
.001	millimeter	mm.	.001	milliliter	ml.	.001	milligram	mg.

Tables showing the comparative values of the Metric and U. S. Systems will be found in the Appendix.

PART III.

GENERAL PROCEDURE OF ANALYSIS.

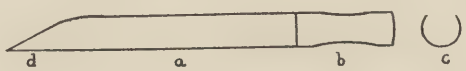
SAMPLING AND PREPARING THE SAMPLE FOR ANALYSIS.

THE object of sampling is to secure such a specimen of the material to be analyzed as will correctly represent the average quality.

Figure 1 represents a convenient sampler or tool for sampling. It consists of a 2-inch brass tube *a*, about 18 inches long, fitted to a handle *b*. It has its surface cut away, giving the section *c*, and the end *d* is rounded or pointed.

If the material to be sampled is in barrels, bags or receptacles of small bulk, the sampler is plunged into each package, and a sample withdrawn. If the contents of a batch barrow are sampled,

Fig. 1.



portions are taken from various parts of the barrow. These different portions are thrown in a heap on a piece of rubber cloth about two feet square, and mixed by raising opposite sides of the cloth. When thoroughly mixed the heap is divided into four parts, by drawing a cross through it, with a large spatula. Two of the quarters are rejected, and the sides of the cloth are again raised, the material mixed, quartered as before, and the opposite quarters rejected again. The process is continued until only enough material remains to fill an 8-ounce, wide-mouth bottle, which is to be tightly stoppered.

If the material is in lumps, it is pulverized before mixing and quartering. This is effected by means of a Mortar and Pestle.

For substances like Coal, an Iron Mortar and pestle are used. (Fig. 2.) For lumpy Soda Ash, Lime, and any substance which

is easily pulverized, and which does not cause wear on the mortar, a mortar and pestle of porcelain or wedgewood are employed. (Fig. 3.) When fine pulverization is necessary, an agate mortar and pestle are made use of. (Fig. 4.) Agate pestles are usually of very short length, and inconvenient to grasp, therefore it is better to fit them into a wooden handle, cementing with a little glycerine and litharge rubbed together into a cream. It is well also to fit the mortar into a stationary wooden block, having a depression to hold it, of such a size as to permit its easy withdrawal for the purpose of emptying.

If the substance is in very hard lumps, such as glass, minerals, etc., a steel mortar and pestle are employed. Figure 5 represents

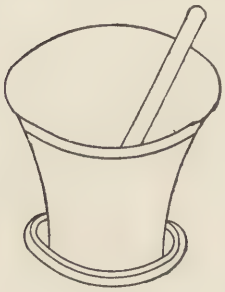


Fig. 2.

Fig. 3.

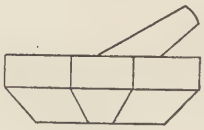


Fig. 4.

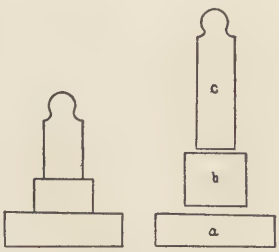


Fig. 5.

this form. The parts *a* and *b* are fitted together. The material to be pulverized is placed in *b*, and the pestle *c* is pushed into *b*. The whole is then held firmly, and *c* is struck with a hammer, driving it into *b*. The mortar is then taken apart, and the contents placed on a clean china plate. A magnet is passed through the fragments to remove any particles of steel abraded from the mortar. The lumps and powder are then removed to an agate mortar for further pulverization.

It is sometimes necessary to dry a powdered sample before weighing it for analysis. This is done by placing the powder in a porcelain dish (Fig. 47), and setting the dish on the shelf of a drying oven (Fig. 37), which is heated to 100° Centigrade. This operation, in individual instances, will be described in Methods of Analysis.

If the substance is in a large shipment, such as a carload, a

number of barrels, etc., a sample is to be taken from every tenth barrel or bag, or at intervals while the car is being emptied, heaped together, and quartered down until it is small enough to be mixed on the rubber cloth as above described.

Sampling must be performed with great care, and the fact must be borne in mind, that the sample is to represent the average quality of the material to be tested, and that an analysis made of an unfair sample is utterly useless as far as practical purposes are concerned.

Some substances, such as batches, are difficult to sample, owing to the different densities of their ingredients. These ingredients becoming separated instead of mixed by the movement, the heaviest sinking to the bottom. It will be found that this trouble may be readily overcome by the use of the rubber cloth.

Where special care is necessary in sampling certain materials, it will be described under the Method of Analysis, which is applicable in such cases.

WEIGHING.

The Balance and Weights.

Figure 6 represents the ordinary form of Analytical Balance.

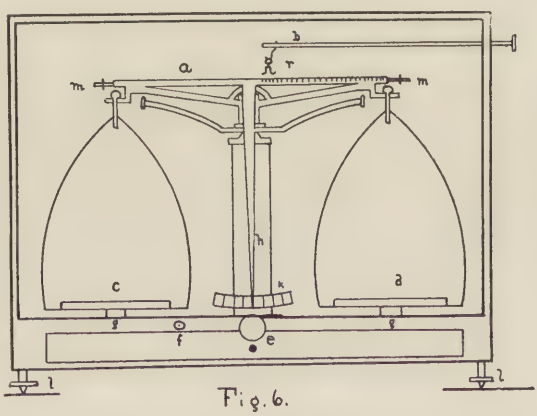


Fig. 6.

The beam *a* is divided into spaces which represent milligrams and fractions when the rider *r* is placed thereon. The rider *r* is moved into position by means of the hooked rod *b*. When not in use the rider is always to be left suspended from the hook.

A pair of watch glasses (Fig. 7) of equal weight are placed on the pans *c* and *d*. The material to be weighed is placed in the watch glass on the pan *c*, and the weights in the watch glass on the pan *d*. The beam *a* is raised and lowered by means of the screw *e*, and the balance finally put into action by pressing the push button *f*, which lowers the pan supports *gg*.

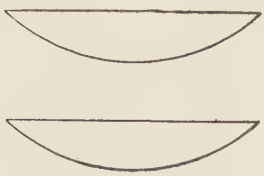


Fig. 7.

When the balance is in equilibrium, the needle *h* swings over equal spaces on each side of the indicator *k*. Sometimes the needle refuses to move, and a gentle current of air must be applied to the pan *d*, by fanning with the hand.

As the balance is a very delicate instrument, and the accuracy of every analysis depends largely on its condition, great care is required in its use. It should never be subjected to any but the most delicate handling. The various parts must be kept free from dust by cleaning with a camel's hair brush, and keeping the door of the glass case closed when the instrument is not in use. No substance should be weighed without the intervention of the watch glasses.

In setting up and adjusting, follow carefully the directions given by the maker. Do not subject the balance to any shock, and do not place it in any position where it will receive vibrations from the building. It is well to have the shelf on which it stands fastened to the wall, and not directly connected with the floor. A good way to avoid vibration is to support the levelling screws *l l* on large rubber bottle-stoppers, having thin disks of metal to prevent the screws from sinking into the rubber. This will obviate much of the vibration. Vibration interferes with the operation of weighing and impairs the sensibility of the balance.

When using the screw *e*, turn it slowly. Each day before beginning work remove, with a camel's hair brush, all dust that may have settled on the beam and pans. Before weighing, see that the balance is correctly adjusted, thus: If the needle *h* does not point to the center of the indicator *k*, adjust it by means of the small screw which is on the pan-rest behind the pan *d*. Turn the screw *e*, and press the button *f*, and if the needle does not move over equal spaces of *k*, release *f*, and turn back *e*, and then

turn the screws *m m*. Again turn *e*, and press *f*, and repeat the process until *h* moves over equal spaces on each side of *k*. It will be readily seen that the side of *k* which shows the greater number of spaces passed over by *h* is the lighter, and the object of the screws *m m* is to overcome this inequality of weight.

Always have in the balance case a small wide-mouth glass vessel containing fused granular Calcium Chloride to assist in keeping the air dry, and preventing any moisture from condensing on the balance. This should be occasionally renewed.

The weights used in chemical analysis are of the Metric system which has been described. (See pages 3 and 4.) These weights are the gram, and its fractions and multiples. A convenient set of weights is shown in Figure 8. It has a range of from 50 grams to 5 milligrams, and contains the following: One of 50 grams, one of 20 grams, two of 10 grams, one of 5 grams, two of 2 grams, one of 1 gram, and also the following fractional weights:

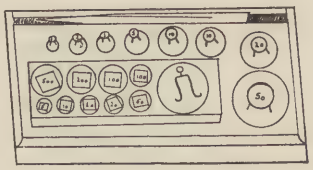


Fig. 8.

One of $\frac{1}{2}$ gram or 0.5 gram or 500 milligrams.
One of $\frac{2}{10}$ gram or 0.2 gram or 200 milligrams.
Two of $\frac{1}{10}$ gram or 0.1 gram or 100 milligrams.
One of $\frac{1}{20}$ gram or 0.05 gram or 50 milligrams.
One of $\frac{1}{50}$ gram or 0.02 gram or 20 milligrams.
Two of $\frac{1}{100}$ gram or 0.01 gram or 10 milligrams.
One of $\frac{1}{200}$ gram or 0.005 gram or 5 milligrams.

Weights lower than 5 milligrams are obtained by the use of the rider *r* on the balance beam. The beam is divided into spaces corresponding to 1 milligram, which in turn, are subdivided into $\frac{1}{10}$ milligrams.

The weights from 50 grams to 1 gram are made of brass, and the fractional weights from 500 milligrams to 5 milligrams of platinum foil.

The sum of the weights used is expressed in grams and decimal fractions, as can be seen in the following example:

One	20	gram weight.
One	10	gram weight.
One	2	gram weight.
One	0.5	gram weight.
One	0.2	gram weight.
One	0.05	gram weight.
	0.0034	gram as obtained by using the rider.

Total weight 32.7534 grams, or 32 grams, $753\frac{4}{10}$ milligrams.

Before replacing the weights in the box, check them by counting up from the vacant spaces, to see if the addition is correct. Each empty space represents a weight taken out and used, and the sum represented by such spaces should correspond to the sum of the weights on the balance pan.

Each weight is to be returned to its proper place in the set, and the set must be kept covered when not in use. The weights should *never* be touched or handled with the bare fingers, but always with the pincers which are provided for this purpose. Always keep the set in a place where it will not be affected by acid fumes. A good place is within the balance case. If the weights become tarnished, do not attempt to brighten them. The crust or film which may form is too slight to seriously affect accuracy. When dusty they may be cleaned with a camel's hair brush.

The weights should be accurately adjusted, that is, they must be correct multiples; for example, the 20 gram weight must be just twice the 10 gram, and twenty times the 1 gram weight, and so on. This point is really of more importance than the absolute accuracy of the weights themselves.

As every analysis depends for its correctness on the quality of the balance and weights, care should be taken to purchase only of a maker of known reputation. It is a very poor plan to sacrifice quality for cheapness in this respect.

Weighing out a portion of the Sample for Analysis.

There are two methods of procedure, namely:

I. By direct weighing.

II. Weighing by difference.

Direct Weighing. Place on the watch glass *d* (Fig. 6) the weight

of the denomination desired, and on the watch glass *c* some of the sample, pouring it from the vial or bottle, or using a three-inch spatula (Fig. 9). Turn the screw *e*, and press the button *f*. If the needle *h* moves towards the right, release *f* and turn back *e*, and remove some of the sample from the watch grass. If on the other hand, *h* moves to the left, add a little more of the sample. Continue thus until *h* moves over the same number of spaces on either side of *k*. The watch glass *c* is then taken from the balance pan and emptied into a convenient receptacle for further treatment. The last portions of the weighed sample are brushed into the same receptacle with a camel's hair brush. The watch glass is then replaced on the balance pan.

Weighing by difference. This method is used when the sample

Fig. 9.

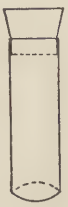


Fig. 10.

is a liquid, or a solid which absorbs moisture when exposed to the air. The sample should be in a glass vial, which has a flat bottom (Fig. 10), and is closed with a cork or glass stopper. Place the vial upright on the watch glass *c*, and on *d* sufficient weights to balance it. Remove the vial, take out the stopper, and pour out a portion of the sample into a convenient vessel. Stopper the vial and replace it on the watch glass. From *d* remove sufficient weights to again balance the vial and contents. The weights removed will represent the weight of the sample taken for analysis.

Example of weighing by difference:

Weight of the vial and contents	25.0392 grams.
Weight after removal of part of the contents	24.0392 grams.
Weight taken for analysis	1.0000 grams.

SOLUTION.

The next step is to bring the weighed sample into solution.

A substance is in solution when it is so diffused through a liquid that its previous form is not apparent to the eye. The liquid is called the *solvent*, and the combination of the body with the solvent is called the *solution*. The solution is named from the material dissolved. A familiar example is that of common salt and water. When salt is placed in water it dissolves or goes into solution, and we have a *solution of salt*. A substance which dissolves readily is said to be *soluble*. When the solvent has no effect on it, it is said to be *insoluble*. When a liquid has dissolved all of the substance that it can, we have a *saturated solution*.

There are three conditions to be noted, viz:

I. The substance may be soluble.
II. It may be insoluble.
III. It may be partially soluble.

I. *The Substance is Soluble.*

The weighed sample being in a suitable vessel, as a beaker (Fig. 11), or a flask (Fig. 12), the solvent liquid is poured in, and if necessary, the vessel is placed on a tripod (Fig. 13), or a

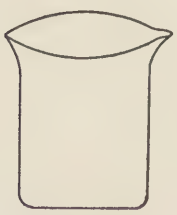


Fig. 11.

Fig. 12.

Fig. 13.

ring stand (Fig. 14), and gently heated by a Bunsen burner (Fig. 15). It is well to interpose a piece of wire gauze between the bottom of the vessel and the flame of the burner. The heating is continued until the sample is dissolved. If effervescence or giving-off of gas is noticed, the beaker should be covered by a watch glass (Fig. 7). When the sample is dissolved the watch

glass is removed, and its convex surface washed with a jet of hot water, by means of a wash bottle (Fig. 20). The washings or drippings should be allowed to run into the beaker. The use of the wash bottle will be considered in detail under Filtering.

II. *The Substance is Insoluble.*

The weighed sample on the watch glass *c* (Fig. 6), is removed to a larger watch glass, and mixed with the proper weight of a suitable flux. This mixture is placed in a platinum crucible (Fig. 16), and the crucible heated by a Bunsen burner (Fig. 15), or a blast lamp (Fig. 17), until the contents are in a fused state. When cool, the fused mass may be dissolved. This will be described in detail under Methods of Analysis.

III. *The Substance is Partially Soluble.*

The weighed sample is treated in a beaker (Fig. 11) with the

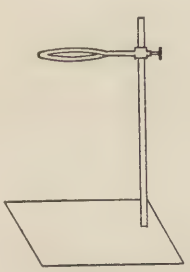


Fig. 14.

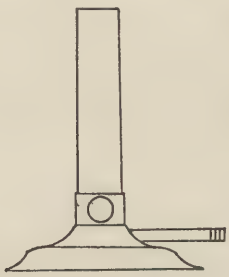


Fig. 15.

Fig. 16.

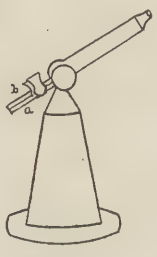


Fig. 17.

solvent liquid, as described under I, the insoluble portion is filtered off (see Filtering), and the filter-paper and contents is heated in a platinum crucible. When the paper has been reduced to ashes, the residue is mixed with a flux, fused, cooled, dissolved, and the second solution added to the first.

PRECIPITATION.

When we have a solution containing an element or compound, which we wish to separate from that solution, we resort to *Precipitation*, and for this purpose we employ what is known as a *Reagent*.

The term *Reagent* is applied to any substance, which when placed in contact with another substance, produces some chemical

change or *reaction*. This is best illustrated by an example. We have, for instance, a solution containing Silver Nitrate ($AgNO_3$). We wish to separate the Silver in some form so that it may be easily weighed. To this solution we add another of Salt or Sodium Chloride (NaCl). The Sodium Chloride *reacts* with the Silver Nitrate, causing a chemical change or *reaction*. The Silver combines with the Chlorine (Cl) and forms Silver Chloride (AgCl), which being insoluble in the surrounding liquid, is deposited or precipitated.

The reaction is written in chemical terms thus:

$AgNO_3 + NaCl = AgCl + NaNO_3$.

Silver Nitrate + Sodium Chloride = Silver Chloride + Sodium Nitrate.

By this means we isolate the Silver Chloride, and can filter it off for further treatment.

In precipitation, the substance which is precipitated is called the *Precipitate*, and the reagent which causes the precipitate to form is called the *Precipitant*.

Precipitates are of different natures, and require different treatment. They can be *flocculent*, when the precipitate floats about in the form of flocks or small clouds. *Granular*, when they are in the form of grains. *Gelatinous*, when they have the appearance of a jelly. *Pulverulent*, when resembling a fine powder. *Crystalline*, when deposited in definite shapes or crystals.

The treatment of the different forms of precipitates will be explained in detail under Methods of Analysis.

FILTRATION OR FILTERING.

To separate a precipitate from a liquid, we filter it, and for this purpose make use of some medium which allows the liquid to pass through it, but retains the precipitate. The best medium is a specially prepared paper known as *Filter Paper*. This paper is made in two qualities, depending on the nature of the operation. They are, Quantitative filter-paper, which is used when it is necessary to preserve the precipitate for further examination, and Qualitative, or Common filter-paper, which is employed when the liquid only is to be further investigated, or when the operation is one in which the presence of the substances and not their proportions are sought.

In connection with the filter-paper we use also a funnel, which is of glass, having the cone (*a*, Fig. 18) made at an angle of exactly 60°, and the stem (*b*) quite long, and ground to a point. It is important that the paper be folded to properly fit the funnel. Fig. 19 shows how this is to be done. Fold the paper in half along the diameter *a*, then again along the radius *b*. Open the

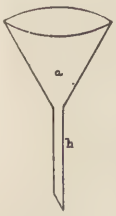


Fig. 18.

Fig. 19.

paper in the form of a cone *c*, having one thickness at *d* and two at *e*. The paper is then placed in the funnel, and pushed down well. While still held by the fingers, the paper is wetted with a jet of water from the wash bottle (Fig. 20). The paper will cling to the funnel, and should be pressed closely to the glass to drive out any bubbles of air. If the funnel is correctly made, the paper will be close to the sides, and the water will run through rapidly.

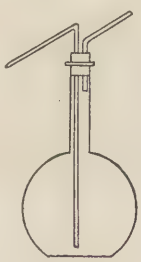


Fig. 20.

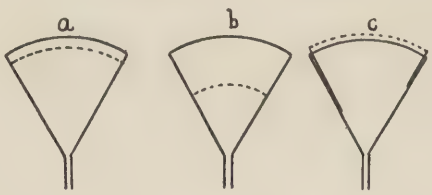


Fig. 21.

Always select a filter-paper of the correct size for the funnel used. It should extend to about an eighth of an inch below the funnel edge. Figure 21 shows the appearance of the paper in the funnel, the cut *a* is the correct way, while *b* and *c* are wrong. The dotted curve shows the upper edge of the paper.

The funnel is then placed in a filter stand (Figs. 22 and 23), the end of the stem being in contact with the inner side of the

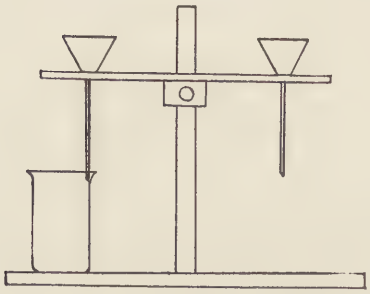


Fig. 22.

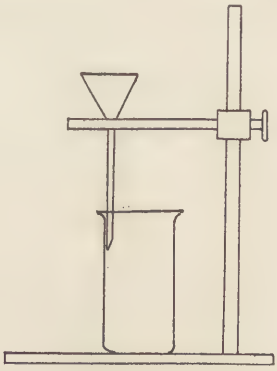


Fig. 23.

glass vessel placed beneath. Hold in one hand the beaker containing the liquid to be filtered, and with the other hold a glass rod against the lip of the beaker (Fig. 24). Incline the beaker and allow the liquid to run gently down the rod into the funnel, at the same time keeping as much of the precipitate as possible in the beaker. Allow the liquid to come to about a quarter of an inch below the edge of the paper, and then stop pouring until the funnel becomes nearly empty. Then continue pouring as before. The object of the glass rod is to prevent loss of liquid or precipitate by running down the outside of the beaker, and also to direct the flow so the liquid will not splash into the funnel. Never allow the liquid to rise over the edge of the filter-paper.

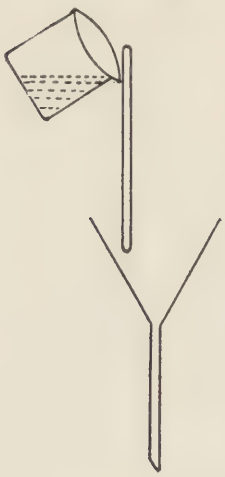


Fig. 24.

When the liquid has all run through the paper, and the beaker is empty, direct a jet of water from the wash bottle (Fig. 20) on to the inside of the beaker. Pour into the funnel as before. Allow the funnel to *entirely* empty, and wash out the beaker as

shown in Figure 25, directing the jet of water so as to wash all the loose precipitate into the funnel. If any precipitate adheres to the beaker or rod, remove it with the aid of another glass rod which has a piece of pure rubber tubing on the end (Fig. 26). This second rod is called a *policeman*, and is used thus: Wet the rubber with a jet of water, and clean the original glass rod by rub-

Fig 25

bing it with the policeman, at the same time holding it over the beaker. When the precipitate has been loosened, place the policeman in the beaker, and wash off the rod into the beaker with a jet of water. Then lay this rod aside.

Rub the inside of the beaker with the policeman, and wash down the loosened precipitate from the sides and bottom. Hold the policeman against the lip, and pour into the funnel. Now direct a jet of water inside the beaker as shown in Fig. 25, washing

the remaining precipitate into the funnel. Wash off the rubber tip of the policeman, allowing the washings to run also into the funnel.

When the funnel again becomes empty, gently direct a jet of water into it, turning the funnel at the same time, so as to wash the precipitate together into the bottom. Care should be taken to

Fig. 26.

have the jet enter the funnel as gently as possible to avoid splashing, as this may cause loss of precipitate. Also begin washing at the edge of the paper, and gradually proceed downward.

After each application of the wash water, allow the funnel to become *entirely* empty before resuming. It is important to wash the precipitate thoroughly. The means of determining when this is properly performed will be given under Methods of Analysis.

Figures 27 and 28 show different kinds of wash bottles. Figure 28 is provided with a piece of rubber tubing *r*, to facilitate the

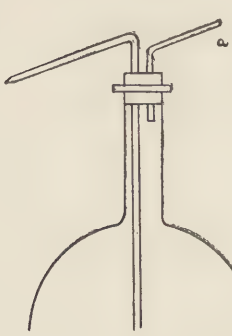


Fig. 27.

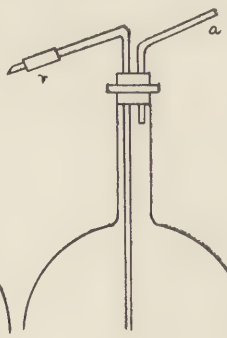


Fig. 28.

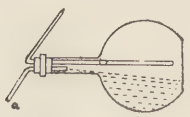


Fig. 29.

directing of the jet. To use the wash bottle grasp it by the neck and blow into *a;* a stream of water is forced out at *b*. When using the flexible tip (Fig. 28), the jet is directed by the forefinger held against the tip *r*.

When a larger stream of water is desired, the bottle is held as in Figure 29, and the water allowed to flow from *a*. When hot

water is used the neck of the wash-bottle is wrapped with cork, sheet asbestos or heavy string to protect the hand.

Terms used in Filtering.

The precipitate remaining on the filter paper is sometimes called the *filter*.

The liquid which runs through the paper is called the *filtrate*.

The liquid which runs through when the filter is washed is called the *washings*.

When a precipitate is so fine as to run through the paper along with the filtrate, it should be filtered through two papers placed one within the other. This will retain most precipitates, but in some cases it may be necessary to pour the filtrate through the paper a second time.

Filtering by Pressure.

Some precipitates, owing to their gelatinous nature, prevent the liquid from passing freely through the paper, and pressure must be used to facilitate the operation.

A cone of platinum foil, pierced with holes (Fig. 30), is placed in the funnel. The paper is folded (Fig. 19) and inserted also in

Fig. 30

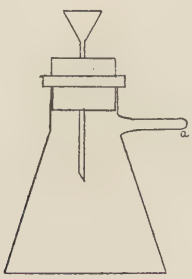


Fig. 31

Fig. 31A

the funnel. The cone prevents the paper from breaking or tearing when pressure is applied. The stem of the funnel is pushed through the hole of a rubber stopper which is inserted in the neck of a filtering flask of heavy glass (Figs. 31 and 31A). A rubber tube is attached to the side tube *a* and connected with an exhausting apparatus. The air drawn through *a* produces a vacuum in

the flask, and consequently a pressure in the funnel, thus causing the liquid to pass rapidly through the paper.

For some purposes a Gooch crucible is used. Figure 32 shows this form. It is made of platinum, and is in three parts. The part *a* has the bottom perforated for filtering. The cover *b* and the

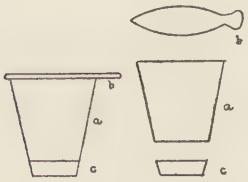


Fig. 32.

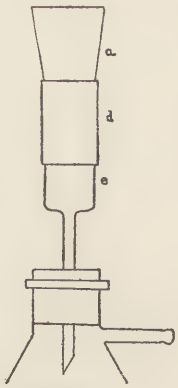


Fig. 33.

cap *c* are placed on the part *a* when the crucible is to be heated. When this crucible is used, the part *a* is inserted in a piece of soft rubber tubing as shown in Figure 33 *d*, and this tubing stretched over a piece of glass tubing *e*, which enters the stopper of the filtering flask. Some fine ignited asbestos is mixed with water and poured into *a* while the pressure is turned on. It packs into a pad or felt on the bottom of *a*, and the crucible is ready for filtering. When the operation is finished, the crucible is removed from the rubber tube, the cap and cover put on, and the contents further treated as required.

To obtain the necessary vacuum we may employ any of the apparatus shown in Figures 34, 35 and 36.

Figure 34 shows a simple contrivance for exhausting. The bottle *b* is provided with an outlet *c* to allow the water to escape. This draws the air through *a* and produces the necessary vacuum. The flow of water from *c* is regulated by the screw compressor *m* which closes the rubber tube *t*.

Figure 35 represents a glass filter pump. The tube *a* is connected with the filter flask by means of a piece of rubber tubing. Another rubber tube at *b* connects the pump with a hydrant.

When the water is turned on it is forced in a jet through the crooked part *c*, drawing the air through *a*. Figure 36 is the same apparatus made of brass, being preferable on account of not being easily broken.

The quality of the filter-paper used in chemical analysis is important. When the precipitate is to be ignited and weighed, care must be taken to use a paper which has the least possible weight of ash. There are excellent filter-papers on the market of both

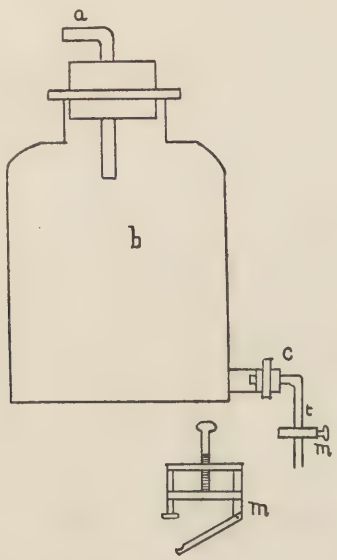


Fig. 34.

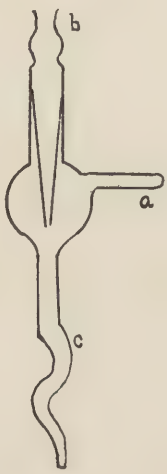


Fig. 35.

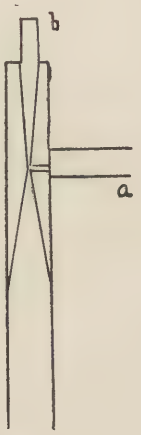


Fig. 36.

German and Swedish make, and while the former is the most advertised, it will be found in the long run that the Swedish is the most uniform in quality. That sold under the name of Munck-tell's Swedish Filter-Paper is perhaps the best for general use, and is of reasonable cost.

When selecting a piece of filter-paper to fit a certain size funnel, always see that the diameter of the paper is a little smaller than twice the diameter of the funnel.

Drying, Igniting, and Weighing the Precipitate.

After the precipitate is thoroughly washed, it is removed with the funnel to a drying oven (Fig. 37). This consists of a copper box with a sheet iron bottom, supported on iron legs, and heated by means of a Bunsen burner placed beneath. The oven has two openings in the top, and a smaller one in one of the sides near the bottom. The lower opening and one of the upper ones are to ven-

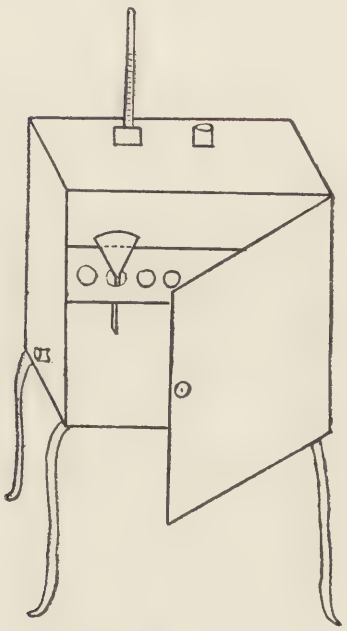


Fig. 37.

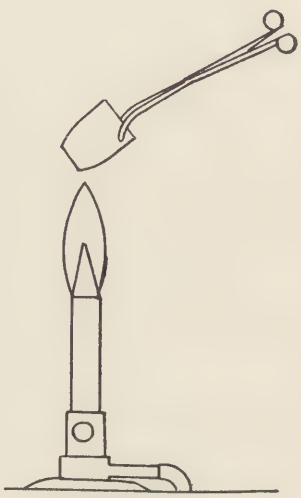


Fig. 38.

tilate the oven, and the other upper opening for the insertion of a thermometer, which is held in place by passing it through a cork. The thermometer should read to 200° Centigrade. The oven is closed by a hinged door, and has a shelf pierced with holes to support funnels.

The funnel is covered by a watch glass or another piece of filter-paper wrapped over it to exclude dirt, etc., and placed in one of the holes of the shelf. The oven door is closed, and the heat

regulated so that the thermometer does not show over 100° C. When the filter-paper and precipitate are dry, the funnel is taken out of the oven, the filter-paper removed, and the precipitate allowed to fall on a small watch glass, a sheet of clean writing paper, or on a sheet of black glazed paper which is sold for this purpose.

A platinum or porcelain crucible with a cover is gently heated by being held with the crucible tongs (Fig. 38) in the flame of a Bunsen burner, and when hot placed in a dessicator (Fig. 39).

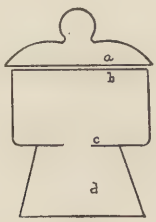


Fig. 39.

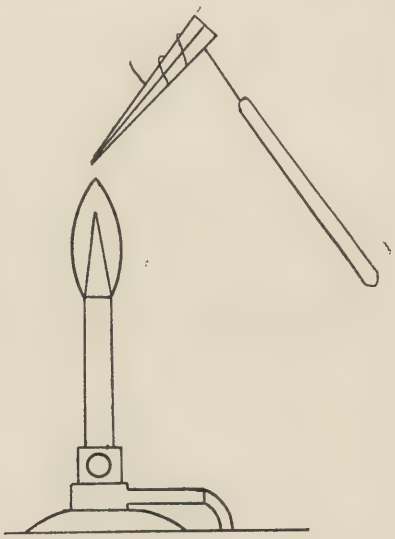


Fig. 40.

When cool, the crucible and cover are weighed on the Analytical Balance, and removed to the glazed paper on which the precipitate has been placed. The filter-paper is then rolled up lengthwise, and held by wrapping round it a piece of platinum wire, which is fused into a glass tube (Fig. 40). The paper is ignited in the burner flame, and allowed to burn while being held over the open crucible. When nothing remains but a charred mass, it is gently detached, and falls into the crucible. The crucible and cover are placed on a clay triangle in an inclined position, and the

burner flame adjusted to play gently against the cover, and be deflected into the crucible (Fig. 41). When the paper is reduced to white ashes the burner is removed.

When cool, the crucible is again placed on a sheet of glazed paper, and the precipitate brushed into it. Any particles that have fallen outside of the crucible are also brushed in. A camel's-hair brush is used for this purpose.

The crucible is then placed on the triangle in an upright posi-

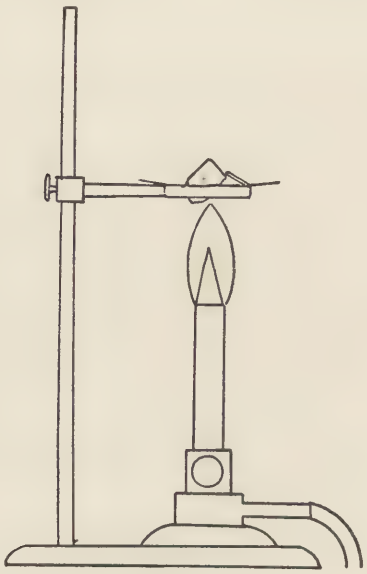


Fig. 41.

Fig. 42.

tion, and partly closed with the cover. A strong heat is applied with the burner. When the contents of the crucible are sufficiently ignited, it is removed together with the cover to the desiccator, and when cool is weighed again.

Example,

Weight of crucible, cover and precipitate	26.9863	grams.
Weight of crucible and cover	26.6381	"
Weight of precipitate	.3482	"

Apparatus used in Igniting and Weighing the Precipitate.

Figure 39 represents the usual form of desiccator. It is of heavy glass, having the under surface of the cover *a* ground to make a tight joint with the edge *b* which is also ground. The narrow part *c* supports a triangle of clay, or of platinum wire to hold the crucible. The lower part *d* contains lumps of fused granular Calcium Chloride, to keep the air in the vessel in a dried state. The object of the desiccator (meaning an apparatus for drying) is to insure the crucible being free from the film of moisture, which collects on all objects which are exposed to the air. This film, while not evident to the eye, may nevertheless, have an appreciable weight. The desiccator also serves to preserve some substances in the crucible from change due to the action of the air, as absorption of water or Carbonic Acid (CO_2).

Figure 16 (page 13) shows a platinum crucible. It may be used for igniting precipitates which are not liable to harm the platinum. It is free from the danger of breakage. It is the crucible always used in the analysis of silicates, where the sample must be brought into a fused state by means of a flux. With proper care, a platinum crucible will last a long time. (See remarks on the care of platinum vessels, Appendix.)

Figure 42 shows a porcelain crucible and cover. Those of Royal Berlin Ware are the best. Porcelain crucibles may be used for nearly all ignitions. They are liable to break when suddenly heated, and the burner flame should be cautiously applied.

Figure 38 shows how to hold a crucible when handling it with the tongs. The tongs are of nickel-plated brass, or of German silver, and sometimes have jaws of platinum.

Figure 43 shows two forms of clay triangles or pipe-stem triangles. They are used with porcelain crucibles. For platinum crucibles, a platinum wire triangle is to be preferred which is shown in Figure 44. The triangle *a* of platinum wire is enclosed within a ring *b* of stout iron wire.

Figure 45 is a Bunsen burner. The gas enters at *a*, and mixes in the tube *b* with air which enters at the hole *c*. This mixture of air and gas passing up the tube *b* is burned at the top. The loose piece *d* is used to regulate the supply of air. When *c* is closed the gas burns with a luminous flame. When *c* is open the flame is blue and of a higher degree of heat. In igniting as described

above, the blue flame should be used, as otherwise a film of carbon will be deposited on the crucible, also, if possible, do not allow the inner flame *e* to touch the crucible.

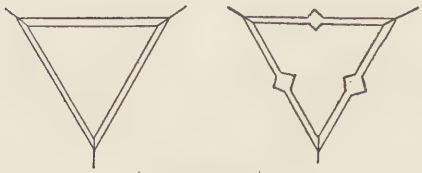


Fig. 43.

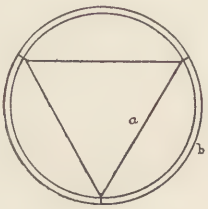


Fig. 44.

When a greater heat than that given by the Bunsen burner is desired, a blast lamp is used (Fig. 17, page 13). The tube *a* is

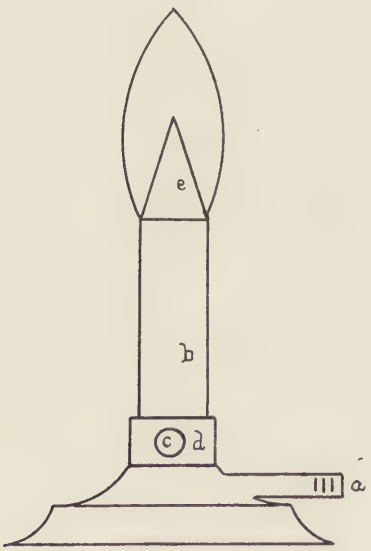


Fig. 45.

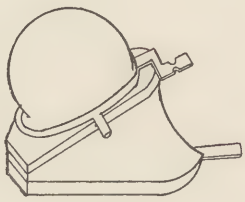


Fig 46

Fig. 47.

connected with the gas supply and *b* with the air supply, obtained from a blower or foot bellows (Fig. 46).

EVAPORATION.

Evaporation is the driving-off of a liquid in which a substance is dissolved.

It is usually conducted in an open vessel, such as a beaker, evaporating dish, or casserole.

Beakers are of thin glass, and are shaped as shown in Figure 11. Evaporating dishes are made of porcelain or platinum. The best porcelain dishes are of Royal Berlin ware (Fig. 47). The smaller sizes are glazed on both sides, and the larger are glazed on the inner side, and partly on the outer. The common German porcelain is usually glazed only on the inner side. Platinum dishes (Fig. 48)

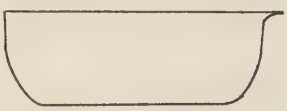


Fig. 48.

Fig. 49.

are used where it is necessary to raise the heat, after the liquid has been driven off.

Casseroles (Fig. 49) are porcelain vessels glazed on both sides, having porcelain handles. They may be either of Royal Berlin Ware or ordinary china. The latter are the cheapest, and are quite efficient for many operations. Casseroles are very useful vessels, and having handles are more convenient than evaporating dishes.

Evaporation is conducted over the flame of a Bunsen burner (Fig. 45), or by the heat of a water-bath, sand-bath, or air-bath. When the direct flame is employed it is well to rest the vessel on a piece of wire gauze or thin sheet-iron to lessen the liability of breaking.

Figure 50 represents a water-bath. It is a copper vessel partly filled with water, and heated by a burner. The top consists of several removable concentric copper rings, to provide different size openings to rest the dishes thereon. The water-bath is used when a gentle heat is required, *i. e.*, the boiling-point of water (100° Centigrade).

Figure 51 shows a sand-bath. It consists of an iron dish filled with sand, and heated with a burner. It is used to obtain a

higher degree of heat than that furnished by the water-bath. The sand-bath is also quicker in its operation, and with care in regulating the burner flame, loss by spattering may be avoided.

Fig. 50.

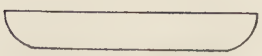


Fig. 51.

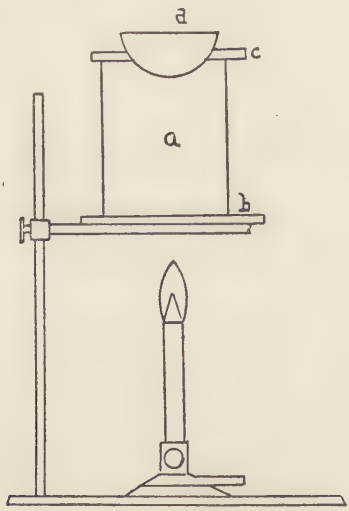


Fig. 52.

Figure 52 represents a simple form of air-bath. It is constructed of a cylinder *a* of iron or other material, open at both

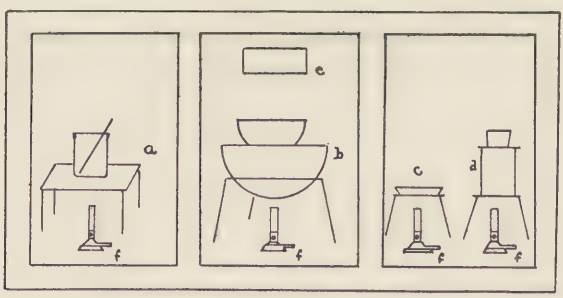


Fig. 52A.

ends and resting on a piece of wire gauze *b*. The upper end is covered by a piece of asbestos board *c* which has an opening in the

center to admit the dish *d*. The air bath is used to obtain a quick, safe heat, and takes the place of the other baths in many operations.

For operations which evolve noxious fumes, the Fume Closet (Fig. 52A) will be found desirable. It consists of a wooden box set against the wall, and connected by means of the opening *e* with the chimney flue of the room. It contains an iron plate *a* supported on four iron legs, the water-bath *b*, the sand-bath *c*, and the air-bath *d*. These are all heated by means of the burners *f*, *f*, *f*, *f*. The closet should be furnished with window sashes, capable of being easily raised or lowered.

PART IV.

VOLUMETRIC ANALYSIS.

VOLUMETRIC analysis is quantitative analysis performed by means of accurately measured volumes of solutions of definite strengths.

It possesses the advantages of great rapidity of operation and the use of simple apparatus.

For successful operation the following are required:

1. A solution whose exact strength is known, called the *Standard Solution.*

2. A solution to enable us to determine when a sufficient volume of the Standard Solution has been used. This is called the *Indicator.*

3. Apparatus accurately graduated, for measuring the proper volume of the standard solution.

The Standard Solution is prepared so that a given volume contains a known weight of the reagent used. The standard solutions mentioned in this book are: Sodium Carbonate, Hydrochloric Acid, Potassium Permanganate, Silver Nitrate, Sodium Chloride, Zinc, Potassium Ferrocyanide.

The indicators employed with the above-mentioned Standard Solutions, will be described with these solutions. The Graduated Apparatus are: Flasks, Burettes and Pipettes.

VOLUMETRIC OR GRADUATED FLASKS.

These are shaped as shown in Figure 53, and may be furnished with a glass stopper or closed with a cork. The former are preferable when the contents have to be mixed by shaking the flask. These flasks are made *to contain* the designated volumes when filled to the mark on the neck at a temperature of 15° Centigrade. A convenient set of flasks consists of the following: 50 Cc., 100 Cc., 200 Cc., 250 Cc., 500 Cc., 1000 Cc., 2000 Cc.

BURETTES.

Figures 54 and 55 represent two forms of Burettes. Figure 54 is a Mohr or pinch-cock burette. It has a glass tip *a* connected with the body of the burette by means of a flexible piece of rubber tubing *b*. The tubing is closed with a pinch-cock *c*. By pressing together the ends of the pinch-cock the rubber tubing opens, and allows the liquid to flow from *a*. This form of burette may be used for all solutions except Potassium Permanganate, as this chemical acts on or corrodes rubber.

Figure 55 is a burette which has a glass stop-cock instead of a

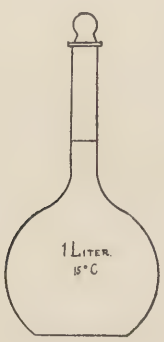


Fig. 53.

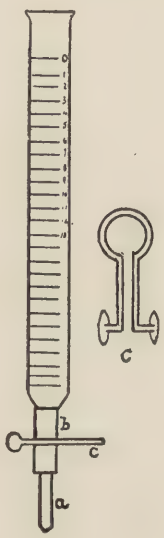


Fig. 54.

Fig. 55.

pinch-cock. This kind may be used for all solutions. It has the disadvantage that the stop-cock is liable to become fast unless well lubricated with vaseline. Liquids which are alkaline, such as Sodium Hydroxide, should never be left overnight in the burette, as they act on the glass and cause the stop-cock to stick.

The burette should be made of a glass tube of even caliber, and accurately graduated. The divisions are either engraved or etched

on the tube. A very convenient burette is one which delivers 50 Cc. and is divided into cubic centimeters and tenths of cubic centimeters.

To use the burette, fill it with the standard solution until the level of the liquid is above the zero mark. Then open the stopcock or pinch-cock and allow the liquid to flow out quickly, closing the cock before the level reaches the zero mark. Then open the cock carefully, and allow the liquid to escape drop by drop until the zero mark is reached. The object of first allowing the liquid to flow quickly is to drive out any air that may be in the narrow part of the burette and cock.

It will be noticed (Fig. 56) that the surface of the liquid assumes the form of a curve, called the *meniscus*, the lower part of

Fig. 56

Fig. 57.

Fig. 58.

Fig. 59.

which is darker than the upper. This dark part must just touch the zero mark, or in other words, the meniscus must be tangent to the mark. This applies also to any mark on the burette.

In using the burette the liquid may at first be delivered one cubic centimeter at a time, the flow being gradually diminished at each addition of the liquid. When the end of the test is approached, it is delivered drop by drop. The test should terminate with a single drop. While adding the standard solution, the liquid in the receiving vessel under the burette must be stirred or shaken gently. The operation of using a burette is called *titration*, from the French word *titre*, meaning *to test* or *vouch*.

In order to insure accurate reading of the burette a float is sometimes used. This is of glass, and shaped as shown in Figure 57. It has a mark which assists the reading. Figure 58 shows a

variety furnished with teeth to keep the float from coming into too close contact with the inside of the burette, and to prevent sticking. Figure 59 shows the appearance of the float in use. To employ the float fill the burette with the liquid and insert the float. The mark should be above the zero mark of the burette.

Fig. 60.

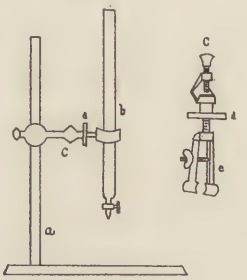


Fig. 61.

Allow enough liquid to escape to bring the float mark on the zero mark. When through titrating observe the mark on the burette with which the float mark coincides. In selecting a float be careful to have it fit the burette not tight enough to stick, nor loose enough to wobble, but so it freely moves up and down without any side play.

PIPETTES.

When it is necessary to add a given volume of a standard solution, *pipettes* are employed. Figure 60 represents a graduated pipette. It is made of glass tubing, and has a mark on the upper, narrow part. When filled to this mark, and the liquid allowed to run out, the pipette *delivers* the proper volume.

To use a pipette insert the lower end in the standard solution, and apply the mouth to the upper end. Gently draw up the liquid until it is above the mark, then quickly close the upper end with the forefinger. Carefully move the finger and permit the liquid to escape until the meniscus reaches the mark. Place the lower end against the inside surface of the receiving vessel and remove the finger. When the flow of liquid stops, the proper

volume has been delivered. When the pipette is properly constructed, it will not be necessary to blow or shake out the last drops. A set of pipettes should consist of the following: 1 Cc., 2 Cc., 5 Cc., 10 Cc., 15 Cc., 20 Cc., 25 Cc., 50 Cc., 100 Cc.

Figure 61 shows the manner in which a burette is supported while being used. The iron stand *a* has the clamp *c*, which rigidly supports the burette *b*. This form of clamp is very useful for supporting apparatus of different kinds. The checknut *d* holds the jaws *e* at any desired angle.

STANDARD SOLUTIONS.

Standard Sodium Carbonate.

Heat a platinum crucible over a burner and remove to a desiccator to cool. When cool place it on the pan *c* of the balance, and on the pan *d* enough weights to counterpoise. Then place on *d* more weights, amounting to about 55 grams. Into the crucible put sufficient c. p. dry Sodium Carbonate to bring the balance again into equilibrium. Place the crucible over a burner and heat for about 15 minutes, adjusting the heat so the bottom of the crucible is a dull red. Remove the crucible to a desiccator, and when cool, place it again on the balance pan *c*. From *d* take away enough weights to balance the crucible and contents. Now remove from the crucible sufficient Sodium Carbonate so that 53 grams remain in the crucible. Dissolve the Sodium Carbonate in distilled water in a beaker, and pour the solution into a graduated liter flask (Fig. 53). Wash out the beaker into the flask. Fill the flask to the mark with distilled water, and mix thoroughly by pouring into a 1500 Cc. *dry* beaker, pouring the solution several times back and forth from the beaker to the flask. Thoroughly wash a glass bottle of about 1 liter capacity, washing it finally with distilled water, and draining as completely as possible. Rinse it out with a little of the Sodium Carbonate solution and drain again. Pour the rest of the solution into it and close with a rubber stopper. Label the bottle thus:

STANDARD SODIUM CARBONATE
Na_2CO_3
53 grams per Liter
1 Cc. = .053 Gms. Na_2CO_3
1 Cc. = 1 Cc. standard HCl.

As this solution is liable to suffer a change of strength on standing, it should be tested occasionally with the Standard Hydrochloric Acid as described below.

Methyl Orange Indicator.

Dissolve about .05 grams of c. p. Methyl Orange in 50 Cc. of distilled water, and keep it in a dropping bottle (Fig. 62). If pure it will dissolve to a bright orange-colored solution. One drop will be sufficient for about 100 Cc. of the solution to be tested. When added to alkaline liquids it imparts a faint yellow color, and to acid solutions a pink tint.

Standard Hydrochloric Acid.

Select some c. p. Hydrochloric Acid having a specific gravity of 1.20. With the hydrometer (see Appendix) verify this specific gravity. Measure out with a graduated cylinder (Fig. 66) about 76 Cc. and pour it into a liter graduated flask (Fig. 53), and fill the flask to the mark with distilled water. Mix thoroughly, as in

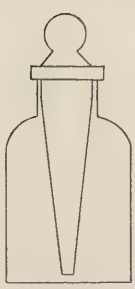


Fig. 62.

Fig. 63.

Fig. 64.

the case of the Standard Sodium Carbonate. Wash out a burette with this acid, and fill the burette with more of it. Bring the level to the zero mark on the burette (Fig. 56). Now fill a 20 Cc. pipette (Fig. 60) to the mark with Standard Sodium Carbonate, and allow it to run into a 100 Cc. Erlenmeyer flask (Fig. 63). Add a drop of Methyl Orange Indicator, and place the flask under the burette. Allow the acid to drop into the flask, observing the

precautions given on page 32, and at the same time shaking the flask with a rotary motion. When a single drop of the acid produces a pink color take the burette reading. If it is found that less than 20 Cc. of the acid is used, then the acid must be diluted so that its strength is the same as that of the sodium carbonate. Suppose we find that 18 Cc. of the acid neutralizes the 20 Cc. of the carbonate, then 900 Cc. of the acid is equal in strength to 1000 Cc. of the carbonate, and we must add 100 Cc. of distilled water to the 900 Cc. of acid in order to equalize the two solutions. The most convenient way to do this is to pour the remaining acid into a mixing jar (Fig. 64), observe the volume, and with a pipette add the proper quantity of distilled water, then insert the stopper and shake thoroughly.

The standard acid is to be kept in a clean glass-stoppered bottle which has been rinsed out with a little of the acid, and labeled thus:

STANDARD HYDROCHLORIC ACID

HCl

1 Cc. = 37 Gms. HCl
1 Cc. = .053 Gms. Na_2CO_3
1 Cc. = .031 Gms. Na_2O.

The Standard Hydrochloric Acid will retain its strength for an indefinite period, and may be used to correct the Standard Sodium Carbonate. The uses of the Standard Acid will be given under Methods of Analysis.

Standard Potassium Permanganate.

Dissolve about 6 grams of c. p. Potassium Permanganate ($KMnO_4$) in distilled water in a beaker. Take care to get all into solution. As the liquid will be very dark in color, it is well to pour it off into a liter flask, and add more water to the beaker, and apply a gentle heat. When dissolved pour the rest of the liquid into the flask and fill with distilled water to the mark.

Now weigh out 0.2 grams of iron piano wire, place it in a 250 Cc. beaker, add 10 Cc. of dilute c. p. Sulphuric Acid (1 part acid to 3 of distilled water) which has been boiled to expel the air. Then add 100 Cc. of hot water, place the beaker under a burette which has been filled with the Permanganate solution, and using the pre-

cautions given on page 32, add enough Permanganate to produce a faint pink color. While adding, stir the contents of the beaker with a glass rod. Read the burette.

Suppose 13.9 Cc. have been used to oxidize the 0.2 grams of iron wire. This weight of this particular kind of wire contains 0.1994 grams of metallic iron, we have

13.9 Cc. $KMnO_4$ equals 0.1994 grams Iron (Fe),

hence 1 Cc. $KMnO_4$ equals 0.0143 grams Fe.

Our solution is too strong, and to reduce it we add water, calculating by means of the following proportion:

$$.0143 : 1000 \text{ Cc.} :: .01 : x \qquad x = 1430.$$

1430 less 1000 equals 430. Therefore to every 1000 Cc. of Permanganate solution we must add 430 Cc. of distilled water, in order to make our standard solution of such a strength that 1 Cc. equals .01 grams of iron. This solution also equals .0143 grams of Sesquioxide of Iron (Fe_2O_3), and .005 grams of Lime (CaO). Keep in a glass-stoppered bottle labeled as follows:

STRONG STANDARD POTASSIUM PERMANGANATE

$KMnO_4$

1 Cc. = .01 Gm. Fe

1 Cc. = .0143 Gms. Fe_2O_3

1 Cc. = .005 Gms. CaO.

When testing for small quantities of Iron a weaker solution of Permanganate is used. It is prepared by taking 100 Cc. of the above, pouring it into a liter flask, and filling with distilled water to the mark. This solution is $\frac{1}{10}$ of the strength of the other. Label the bottle as follows:

WEAK STANDARD PERMANGANATE

1 Cc. = .001 Gm. Fe

1 Cc. = .0014 Gm. Fe_2O_3.

Standard Silver Nitrate.

Dissolve about 17 grams of c. p. Silver Nitrate ($AgNO_3$) in distilled water in a beaker. Pour into a liter flask and fill with distilled water to the mark. To correct this solution we require one of Sodium Chloride the value of which is known. To make up this latter solution proceed as follows: Place about 2 grams of c. p.

Sodium Chloride (NaCl) in a crucible and heat sufficiently to cause the salt to fuse. Carefully weigh out 1 gram of the fused salt, dissolve it in distilled water, pour into a liter flask and fill to the mark. Keep in a glass-stoppered bottle labeled thus:

STANDARD SODIUM CHLORIDE
NaCl
1 gram per liter
1 Cc. = 0.000606 Gm. Cl.

Dissolve about 10 grams c. p. Potassium Chromate (K_2CrO_4) in 100 Cc. of distilled water, place it in a bottle and label it POTASSIUM CHROMATE INDICATOR. With a pipette carefully measure 100 Cc. of the Silver Nitrate solution and allow it to run into a porcelain dish (Fig. 47). Add a drop of the Potassium Chromate Indicator, and from a burette add sufficient Silver Nitrate solution to produce a faint reddish color. While adding, stir constantly with a glass rod, and observe the precautions given on page 32. Carefully note the red color produced, and in subsequent operations stop adding the Silver Nitrate when the same tint is noticed as when adjusting the Standard Silver Nitrate.

Knowing the strength of 100 Cc. of the Standard Sodium Chloride, we can calculate the value of the Standard Silver Nitrate Solution. Suppose we found that 17 Cc. of the Silver Nitrate solution produced the red color in the 100 Cc. of Sodium Chloride solution, we have,

17 Cc. Silver Nitrate equals 100 Cc. Sodium Chloride
or 17 Cc. = .0606 grams Chlorine (Cl).
therefore 1 Cc. = .00356 grams Chlorine (Cl).

Label the bottle containing the Silver Nitrate solution as follows:

STANDARD SILVER NITRATE
$AgNO_3$
17 grams per liter
1 Cc. = .00356 Gm. Cl
1 Cc. = .0059 Gm. NaCl.

Standard Zinc Solution.

Weigh 10 grams of c. p. metallic zinc in stick form, and place it in a liter flask. Add about 100 Cc. dilute c. p. Hydrochloric

Acid (1 part acid, 3 parts water). When the Zinc has dissolved fill the flask with distilled water to the mark and mix well. Pour this solution into a glass-stoppered bottle, and label thus:

STANDARD ZINC
10 grams Zn per liter
1 Cc. = .01 Gm. Zn
1 Cc. = .01246 Gm. ZnO.

Standard Potassium Ferrocyanide Solution.

Weigh out 43 grams of c. p. Potassium Ferrocyanide ($K_4Fe(CN)_6$). Place it in a liter flask, and dissolve in distilled water. Fill to the mark and mix well. Pour into a glass-stoppered bottle, and label as follows:

STANDARD POTASSIUM FERROCYANIDE
$K_4Fe(CN)_6$
43 grams per liter.

This solution must be corrected by means of the Standard Zinc as follows: Carefully measure 10 Cc. of the Standard Zinc Solution with a pipette, and allow it to run into a 50 Cc. beaker. Cautiously add the Zinc Solution from a burette. A precipitate will form which will at first be coarse, but which will gradually get finer as more Zinc Solution is added. As the precipitate gets fine take out a drop of the solution with a glass rod, and place it on a china plate in contact with a drop of strong Uranium Acetate solution (5 Gm. to 100 Cc. water). When a brown color appears on the plate the test is finished. Observe the volume of the Ferrocyanide Solution used, and calculate its value in terms of Zinc. As the Ferrocyanide changes in strength, it must always be tested before use as described above, and the value calculated.

PART V.

REAGENTS.

THE balance used in weighing out the various reagents is of the Robervahl type (Fig. 65). When new it is sensitive to about a $\frac{1}{2}$ gram and will turn with a load of 1 kilo. The weights most convenient to use are Metric block weights of the following de-

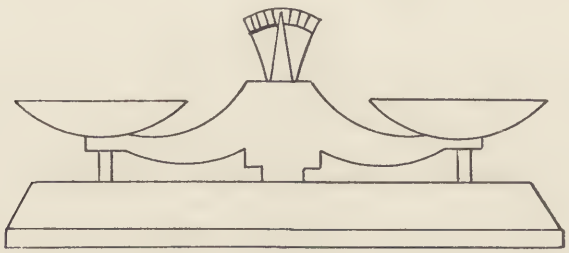


Fig. 65.

Fig. 66.

nominations: 100 Gms., 50 Gms., 20 Gms., two 10 Gm. weights, 5 Gms., two 2 Gms. and one 1 Gm. They are similar in shape to those shown in Figure 8.

Figure 66 represents a graduate measure of cylindrical shape. It is well to have four of these of the following capacities: 1000 Cc., 500 Cc., 250 Cc., 100 Cc.

Distilled Water. H_2O.

When the word *Water* is employed in this book, *Distilled Water* is meant. Distilled water is made by evaporating any water and condensing the steam. Figure 67 represents the usual type of still employed, *a* is a tin-lined copper boiler, fitted with a head *b* of tin-lined copper. This head with the beak *c* is detachable from *a* for convenience in cleaning and filling. The beak *c* is connected by a union joint *d* with the worm *e*. The worm *e* is of block tin

and passes through the zinc condenser *f*. Hydrant water is allowed to pass into *f* through a rubber tube to the bottom. The water cools the worm *e* and escapes at *g*. Water to be distilled is

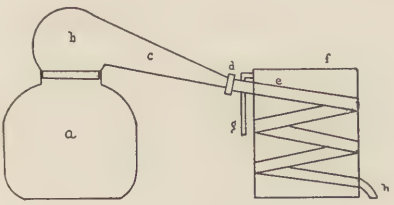


Fig. 67.

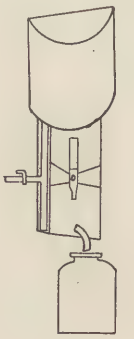


Fig. 68.

poured into *a*. Heat is applied to the bottom of *a*, and the steam is condensed in *e*, the distilled water being collected at *h*.

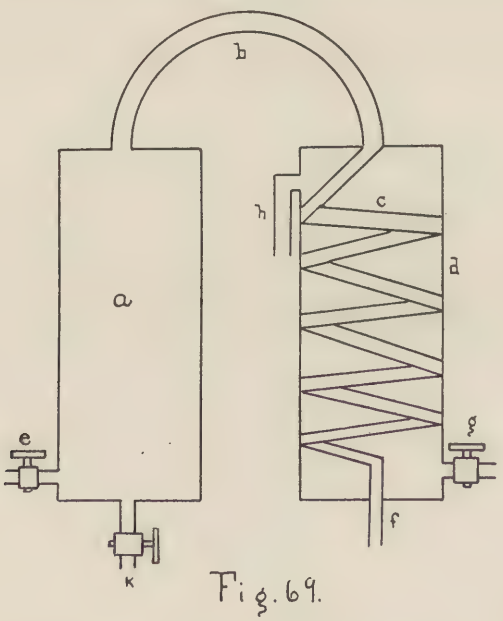


Fig. 69.

Figure 68 represents the Domestic Water Still. This has the

advantage of being continuous in its operation, and requires little attention.

Figure 69 shows a contrivance for obtaining distilled water from the waste steam of a factory boiler. The steam enters the iron cylinder *a* through the cock *e*, it passes through closely packed gravel with which *a* is filled, and enters the block-tin pipe *b* which is continued in the worm *c* which is also of block tin. Cold water to cool the worm *c* is admitted to the iron cylinder *d* by means of the cock *g*. It fills *d* and escapes at *h*. The condensed steam is collected at *f*. The cylinder *a* is emptied of any water of condensation by means of the cock *k*.

Distilled water should have no odor, and exhibit no reaction when tested with litmus paper (see page 51). It should give no precipitate when Silver Nitrate or Barium Chloride is added, and no residue when evaporated to dryness.

ACIDS.

Sulphuric Acid. H_2SO_4.

There are two kinds employed, the commercial, and the chemically pure. The former is used for the evolution of gases, such as Carbon Dioxide, Sulphuretted Hydrogen, etc. The chemically pure acid should be colorless, and should give no odor when shaken. When a colorless solution of ferrous sulphate is poured into it, the liquids in contact should give no brown tint. When poured on pure zinc, and the resultant gas passed through a glass tube, and burned, and the flame allowed to touch a clean porcelain dish, no metallic film must be deposited. To dilute add *cautiously* one part of acid to five parts of water.

Nitric Acid. HNO_3.

Pure Nitric Acid is colorless, and leaves no residue when evaporated in a platinum dish. When diluted with water, and a solution of Silver Nitrate or Barium Chloride is added, no turbidity should appear. On standing for a time Nitric Acid becomes yellow in color, this may be removed by blowing air through the acid. Dilute Nitric Acid is made by adding an equal volume of water.

Hydrochloric Acid. HCl.

There are two kinds used, the commercial and the chemically pure. The former is used for the evolution of gases. The latter should be colorless, and when evaporated leave no residue. It should remain colorless during evaporation. When mixed with water and a solution of Barium Chloride added, no precipitate or turbidity should appear. When neutralized with Ammonia, and Ammonium Sulphide added, no precipitate should form. To dilute add one part of acid to two parts of water.

Nitro Hydrochloric Acid. Aqua Regia.

This is formed by mixing 1 part of Nitric Acid with 3 parts of Hydrochloric Acid. This reagent is not to be used diluted.

Hydrofluoric Acid. HF.

This acid when pure must leave no residue when evaporated in a platinum dish. As it attacks glass it is put up in Cerasine bottles.

Acetic Acid. $C_2H_4O_2$.

This acid should have a specific gravity of 1.04. It should leave no residue on evaporation, and give no precipitate when tested with Hydrogen Disulphide, Silver Nitrate, or Barium Chloride. When neutralized with Ammonia and Ammonium Sulphide added, it should not become cloudy.

Bromine. Br.

Bromine is a dark brown liquid of specific gravity 2.97. It is very corrosive, and the fumes should not be breathed or allowed to come in contact with the eyes. It is to be kept in a glass-stoppered bottle furnished with a ground-glass cap. A solution known as Bromine Water is used, made by placing about 25 Cc. in an 8-oz. bottle, and filling the bottle with water and shaking. As the solution is used up, more water is added.

GASES.

Carbon Dioxide. Carbonic Acid. CO_2.

This gas is formed when a carbonate is acted on by an acid. Calcium Carbonate (Chalk, Marble, Limestone) and Sulphuric or

Hydrochloric Acid are commonly used. Figure 70 shows a simple apparatus for producing this gas. The flask *a* is fitted with a two-hole rubber stopper *b* through which passes the funnel *c* and the tube *d*. The tube *d* is bent to enter the bottle *e* which contains water to wash the gas, the gas escaping through the tube *f*. Figure 71 represents a Kipp Gas Generator which is used when quantities

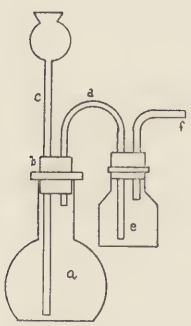


Fig. 70.

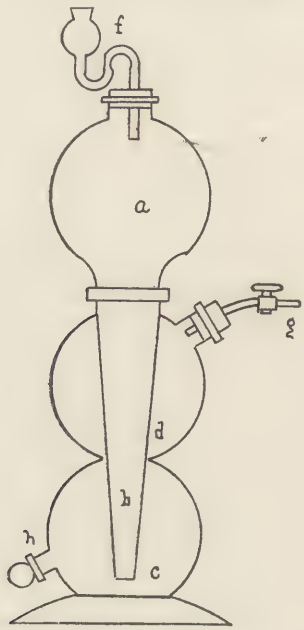


Fig. 71.

of gas are required. The upper part *a* is extended into a tube *b* which reaches almost to the bottom of *c*. The part *b* should be of such a diameter as to barely pass through the bottom of *d*. The substance acted upon by the acid is placed in *d*, and the acid poured through the funnel *f* into *c* until it rises in *d*. The gas escapes at *g*. The spent acid may be drawn off at *h*.

Hydrogen Sulphide. H_2S.

This gas is formed by the action of Sulphuric or Hydrochloric Acid on Ferrous Sulphide. Either of the apparatus described

above may be used. For adding small volumes of this gas to solutions, the apparatus described in Figure 72, designed by Dr. A. H. Elliott, will be found very convenient. The Ferrous Sulphide is placed in the 8-drachm homeopathic vial *a* fitted with a one-hole rubber stopper *b* through which passes the tube *c* which is enlarged to admit a plug of cotton in *d*. A bent tube *e* is fitted into a cork *f* which is inserted in *d* and delivers the gas.

Hydrogen Sulphide is also used in solution. This is made by

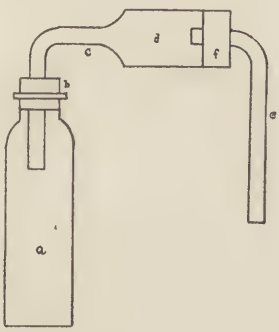


Fig. 72.

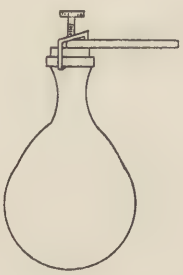


Fig. 73.

passing the gas through distilled water, until strongly saturated which may be perceived by the odor given when the solution is shaken.

Hydrogen. H.

This gas is formed when Zinc is acted on by Sulphuric or Hydrochloric Acid. The apparatus described under Carbonic Acid may be employed.

Oxygen. O.

Oxygen may be made by heating a mixture of 100 parts Potassium Chlorate and 5 parts Manganese Dioxide, in a copper retort (Fig. 73). The gas thus produced may be collected in rubber gas-bags (Fig. 74). For laboratory purposes it is best to purchase the gas compressed in cylinders, which may be procured in various sizes.

Fig. 74.

ALKALIES AND ALKALINE SALTS.

Sodium Hydroxide. $NaOH$.

This reagent may be purchased in a sufficiently pure state. It comes in the form of sticks and must be kept from the air. It is best preserved in a wide-mouth, glass-stoppered bottle.

Potassium Hydroxide. KOH.

This is in the same form as Sodium Hydroxide, and must be preserved with the same precautions.

Ammonium Hydroxide. NH_4OH.

This is sold as Stronger Water of Ammonia, and has a density of .90. The solution used in ordinary laboratory operations is formed by adding one part of distilled water to one of ammonia. It should be colorless and leave no residue when evaporated to dryness in a platinum dish. An equal volume of lime water, added to the diluted Ammonia should cause but slight turbidity.

Ammonium Sulphide. $(NH_4)_2S$.

This is formed by passing Hydrogen Disulphide through dilute Ammonia until the latter is saturated. It should be kept in glass-stoppered, amber-colored bottles.

Sodium Carbonate. Na_2CO_3.

The dry salt is used. It should be perfectly white, and should dissolve in water without leaving any residue. When the solution is made acid with Nitric Acid and Barium Chloride added, no precipitate or turbidity should form. When tested in the same manner with Silver Nitrate, no turbidity should appear. When evaporated to dryness with Hydrochloric Acid, re-dissolved in Hydrochloric Acid, and again evaporated and re-dissolved, no insoluble residue should remain.

Potassium Carbonate. K_2CO_3.

This salt should also be procured in the dry state, and tested for impurities in the same manner as Sodium Carbonate.

Flux for Silicates.

Mix in a clean dry porcelain dish 53 parts by weight of Sodium

Carbonate, and 70 parts by weight of Potassium Carbonate, and keep in a glass-stoppered, wide-mouth bottle. This flux is used to effect fusion in the analysis of glass and other silicates.

Ammonium Carbonate. $(NH_4)_2CO_3$.

This salt is in the form of transparent crystals. When exposed to the air a white crust forms, which should be scraped off before dissolving. The pure salt should entirely volatilize when heated. After adding Nitric Acid in excess to a solution of Ammonium Carbonate no precipitate should form when tested with Hydrogen Sulphide, Silver Nitrate or Barium Chloride. When used as a reagent one part by weight is dissolved in a mixture of water (four parts) and ammonia (one part).

Ammonium Chloride. NH_4Cl.

This is referred to under J. Lawrence Smith's method for the determination of Alkalies in Silicates (see page 85).

Ammonium Oxalate. $(NH_4)_2C_2O_4.2H_2O$.

It is used in the form of white crystals. The solution should remain clear when tested with Hydrogen Sulphide, and the salt should entirely volatilize when heated on platinum. For use dissolve one part by weight in twenty-four parts of water.

Sodium Ammonium Phosphate. $NaNH_4PO_4.4H_2O$.

This is also known as Microcosmic Salt. It is to be dissolved in six parts cold water, but should not be kept in solution, as it acts on glass. It should be made up as required.

Hydrogen Di-Sodium Phosphate. $Na_2HPO_4.12H_2O$.

It is also called Sodium Phosphate. It should be dissolved as required, as it attacks glass. The proper solution is one part in ten of water.

Potassium Nitrate. KNO_3.

This is in the form of white crystals, and may be procured in a sufficient state of purity.

Sodium Nitrate. $NaNO_3$.

This salt is similar in properties and uses to the above.

Potassium Bichromate. $K_2Cr_2O_7$.

An orange-colored crystalline salt, referred to in this book under Volumetric Analysis.

Potassium Bisulphate. $KHSO_4$.

A white crystalline salt which, when dissolved in a large excess of water, decomposes into Potassium Sulphate and Sulphuric Acid. When heated it gives off first its combined water, and when the heat is raised to redness Sulphuric Acid escapes.

Sodium Bisulphate. $NaHSO_4$.

Similar in appearance and properties to the above.

Potassium Ferrocyanide. $K_4Fe(CN)_6$.

A yellow crystalline salt which is used in testing for Iron. It gives a blue coloration of ferrocyanide of iron (Prussian Blue) when *ferric* iron is present. For use as a reagent dissolve one part of the salt in twelve of water.

Potassium Ferricyanide. $K_3Fe(CN)_6$.

A blood-red crystalline salt used for testing the presence of *ferrous* iron, with which it produces a blue color. Ferric salts produce no color. Should be made up as wanted and not kept in solution.

Potassium Permanganate. $KMnO_4$.

This salt is in the form of purple-colored, needle-shaped crystals. It is referred to in this book under Volumetric Analysis. It is readily obtained in a pure state.

Potassium Thiocyanate. $KCNS$.

A colorless crystalline salt. When used as a reagent one part is dissolved in ten parts of water. When Hydrochloric Acid is added to this solution it should remain colorless. It is used as a test for very small quanties of iron in the *ferric* state. When added to a ferric solution a deep-red color is produced.

Sodium Chloride, Salt. $NaCl$.

This is used to prepare the Standard Salt Solution, page 38.

The best grades of table salt being very pure, are suitable for use as this reagent, provided the solution is not precipitated or ren-

dered turbid by Ammonium Oxalate, Sodium Phosphate, or Barium Chloride.

Soda Lime.

This is referred to under Methods of Analysis, Lime. It is an absorbent for Carbon Dioxide (CO_2) and Water (H_2O). It is purchased in the form of brownish lumps.

ALKALINE EARTH SALTS.

Barium Chloride. $BaCl_2.2H_2O$.

A white crystalline salt used in the determination of Sulphur. It is to be dissolved in the proportion of one part to ten of water.

Calcium Chloride. $CaCl_2$.

It is used as a reagent and as an absorbent for hygroscopic moisture. In the latter case it is in the form of porous lumps. See Desiccator, General Procedure of Analysis.

Calcium Carbonate. $CaCO_3$.

This reagent is referred to in this book under Determination of Alkalies in Silicates, in the method of J. Lawence Smith. When used for this purpose it should be in the form of a white powder, which should not give any blue color when shaken up in water and a drop of litmus added. This shows the presence of alkalies, and is peculiar to Calcium Carbonate, which has been made by precipitation with Sodium Carbonate and not properly washed. It may be purified by placing on a filter-paper in a funnel, and thoroughly washing with hot water.

Magnesia Mixture.

This reagent is referred to on page 75, Determination of Arsenic. It is made by dissolving 55 grams of crystallized Magnesium Chloride in water and filtering, then dissolving 14 grams of Ammonium Chloride in water, adding a little Bromine water and an excess of Ammonia, and filtering. This second solution is added to the first, more Ammonia is added to give a decided odor, and the solution diluted to about a liter, transferred to a bottle and shaken well. When required a sufficient quantity is passed through a filter and used.

METALS AND METALLIC SALTS.

Iron Wire. Fe.

This is used to standardize the Potassium Permanganate Volumetric Solution (page 36). It may be obtained from dealers in chemicals, and before being weighed should first be cleaned with emery-paper and then wiped with filter-paper.

Ferrous Sulphate. $FeSO_4.7H_2O$.

This is used in qualitative analysis in testing for nitrates. It is in the form of pale green crystals, which, when dissolved in water, must leave no brownish-yellow Ferric Sulphate undissolved in the solution. When Hydrochloric Acid is added to the solution, and Hydrogen Sulphide gas passed into it, no precipitate or black tint must be produced.

Cupric Sulphate, Anhydrous. $CuSO_4$.

Heat crystals of C. P. Copper Sulphate in a porcelain crucible until the combined water is driven off, that is, until they turn white. This salt is used in the U tube of the CO_2 apparatus, described on page 64, for the absorption of any chlorine that may be liberated.

Platinic Chloride. $PtCl_4$.

It is used to separate Potassium from Sodium in the analysis of glass, clay, etc. It can be purchased, or may be made from scraps of metallic Platinum which accumulate in the laboratory. The following method of Fresenius may be employed: Place about five parts by weight of metallic zinc in a clay crucible and cover with some common salt to prevent oxidization. Heat until the zinc is melted, and add little by little one part of the platinum scraps. When the last addition is melted remove the crucible, and when cold dissolve the alloy formed in dilute Hydrochloric Acid. When effervescence has ceased add more acid and boil for a short time. Pour off or filter the platinum which is left, and thoroughly wash with water. Boil it in nitric acid and wash again. Dissolve in Aqua Regia and evaporate on the water-bath, adding a little Hydrochloric Acid from time to time. When in a syrupy condition dissolve in ten parts of water.

Platinic Chloride may also be made from the washings which

come from the separation of Potassium and Sodium (page 85) by the following method: The solution containing the platinum salts is boiled in a beaker, and sufficient Ferrous Sulphate added to give it a greenish tint. Then add about one gram of granulated sugar and boil for a short time. Filter off the precipitated Platinum Black and wash with hot water. Remove to a casserole and gently heat with Hydrochloric Acid. Pour off the acid, keeping the platinum black in the casserole, and wash three times with hot water, pouring off the water each time. Add a little Nitric Acid and heat again. Then add more Hydrochloric Acid and evaporate on the water-bath until the mass is of the consistency of syrup. Dissolve in ten parts of water for use.

Silver Nitrate. $AgNO_3$.

This reagent is in the form of white crystals which must be kept in a stoppered bottle of dark color, to prevent the action of light. For use dissolve 1 part of the crystals in 20 parts of water. To test the solution for purity add Hydrochloric Acid until no more precipitate forms. Filter, and pass Hydrogen Sulphide gas into the filtrate; no precipitate or turbidity should appear.

Zinc. *Zn.*

Metallic Zinc for analysis may be purchased chemically pure in the form of sticks. It is used to reduce iron to a ferrous state, and in the preparation of hydrogen gas.

LITMUS PAPER.

This reagent is an indicator used to determine whether a solution is acid or alkaline. It may be purchased in sheets and cut into strips. It should be kept in a glass-stoppered bottle, so as not to be affected by the fumes of the laboratory. The paper is colored either red or blue. Acid solutions will change the color of the blue paper to red, and alkalies will restore the color or turn the red paper blue.

TURMERIC PAPER.

This is of a brownish-yellow color, and is used to test for the presence of Boron compounds (Borax, etc.), which cause it to assume a reddish tint.

Reagents in solution should be kept in glass-stoppered bottles,

properly labeled. A good form of reagent bottle is shown in Figure 75. It has the advantage of an indestructible label, the name and symbol of the reagent being blown in the glass. It is sold at a reasonable price. Paper labels should be avoided if possible on reagent bottles, as they are being continually destroyed by the liquid running down the bottle. When they are unavoidable they should be covered with a thin coat of paraffin. A convenient size of reagent bottle is 8 ounces, or 250 Cc.

FIG. 75.

PART VI.

METHODS OF ANALYSIS.

SODA ASH.

Sodium Carbonate, Carbonate of Soda, Na_2CO_3.

Total Alkali or Oxide (Na_2O), Alkalinity or Test Degree.

WEIGH out 3.1 grams and brush it into a 50 Cc. beaker. Add about 25 Cc. hot water and boil until the soluble part has been dissolved. Filter through a 9 Cm. paper into a 100 Cc. Erlenmeyer flask (Fig. 63) and wash the filter three times with hot water. To the liquid in the flask add a drop of Methyl Orange Indicator, and place the flask beneath a burette which contains the standard Hydrochloric Acid, prepared as on page 35. Cautiously add the Standard Acid, 1 Cc. at a time, until about 40 Cc. are in, shaking the flask at the same time with a rotary motion. Then add more Standard Acid, a few drops at a time, and as the test approaches the end, one drop at a time. When a single drop produces a full pink color the test is finished. The number of Cc. and tenths of acid used will be the degree of alkalinity or per cent. of Na_2O in the ash. This number multiplied by 1.71 will give the per cent. of carbonate or Na_2CO_3. Example:

Weight of Soda Ash taken for analysis,	3.1 grams.
Cc. Standard Acid used,	46.0 Cc.
Per cent. of Na_2O or test degree,	46.0 per cent.
Per cent. of Na_2CO_3,	78.66 "

In case the results are wanted quickly the solution in water and filtering may be dispensed with as follows: Weigh 3.1 grams of the ash and brush it into an Erlenmeyer flask. Add about 10 Cc. of cold water and shake gently. Add a drop of Methyl Orange Indicator, and remove to the burette and allow the acid to drop in

as described above. The results will be slightly high on account of the insoluble lime compounds using up some of the acid.

DETERMINATION OF SALT, OR SODIUM CHLORIDE.

Salt is present in Soda Ash as an impurity or adulterant. It is sometimes added to 58 per cent. ash to reduce the strength to 48 per cent.

Weigh out 1 gram of Soda Ash, brush it into a 25 Cc. beaker, and add 10 Cc. of hot water. Cover the beaker with a watch-glass, and from a smaller beaker add carefully 5 Cc. of dilute Nitric Acid. While adding the acid keep the watch-glass on the beaker to avoid loss by effervescence. Boil gently until effervescence ceases. Remove from the burner, and with the wash-bottle rinse off the watch-glass, allowing the rinsings to fall into the beaker. Filter through a 7 Cm. filter-paper into a 50 Cc. beaker, and wash the first beaker into the funnel, using a jet of hot water from the wash-bottle. Wash the filter three times with hot water. Place the beaker over a burner and heat gently to boiling. While boiling add about 10 Cc. of Silver Nitrate Solution. While adding this reagent keep stirring the boiling solution with a glass rod. Continue stirring and boiling until the precipitated Silver Chloride settles, leaving the liquid above it clear. Remove the beaker from the burner and set it aside to settle thoroughly. Filter through a 11 Cm. ashless filter-paper and wash with hot water, observing the precautions given on pages 16 to 18 regarding the complete removal of the precipitate from the beaker. Wash with hot water until the washings give no residue when the last few drops are evaporated on the cover of a platinum crucible. Place the funnel containing the filter-paper in an air-bath, and when dry burn the paper as directed on page 23. Ignite in a weighed porcelain crucible and allow to cool. When cool add a drop of strong Nitric Acid, and gently heat by moving the burner to and fro beneath the crucible, taking care not to allow the acid to spatter. When dry ignite strongly until the contents of the crucible are white. Remove to a desiccator, allow to cool, and weigh. The weight of the Silver Chloride multiplied by 40.85 will give the per cent. of Salt or Sodium Chloride in the Soda Ash.

Example:

Weight of Soda Ash taken, 1 gram.

Weight of crucible, cover and Silver Chloride,	35.9873
Weight of crucible and cover,	35.6392
Weight of Silver Chloride (AgCl),	.3481

$.3481 \times 40.85 = 14.22$ per cent. of Salt, NaCl.

NOTE: The object of adding the Silver Nitrate when the solution is boiling is to insure precipitation in a granular form, thus causing it to settle quickly. If the chloride comes down in a fine precipitate it is liable to run through the filter-paper.

RAPID METHOD FOR SODIUM CHLORIDE.

Dissolve 1 gram of Soda Ash in 25 Cc. of water in a porcelain dish, cover the dish with a watch-glass, and add sufficient strong Nitric Acid to make a slight excess, that is, to turn litmus-paper red. Boil gently until no more bubbles of Carbonic Acid escape, then add little by little dry c. p. Sodium Carbonate until the Litmus paper is blue when placed in the solution. Remove the watch-glass and wash it with a jet of cold water. Place the dish beneath a burette which contains standard Silver Nitrate. To the contents of the dish add two drops of Potassium Chromate indicator, and allow the standard solution to drop in, proceeding exactly as described on page 38. When the red color is produced the test is finished.

Example:

1 gram Soda Ash.

Silver Nitrate used 24.1 Cc. $\times$ $(.0059 \times 100) = 14.219$ per cent. Salt, NaCl.

Determination of Salt Cake or Sodium Sulphate. Na_2SO_4.

This may be present as an impurity in Ash which has been made by the Le Blanc process, or it may have been purposely added to Ash to be used for Amber glass.

Weigh 5 grams of Soda Ash into a 25 Cc. beaker. Add 10 Cc. of hot water and 5 Cc. of strong c. p. Hydrochloric Acid, at the same time keeping the beaker covered with a watch-glass, and adding the acid gradually from a smaller beaker. Boil gently for a few minutes. Remove the watch-glass and wash its surface with a jet of hot water, allowing the washings to run into the beaker.

Filter through a 7 Cm. filter-paper into a 50 Cc. beaker, and wash out the first beaker with a jet of hot water, allowing the washings to run into the funnel. Wash the filter three times with hot water. Bring the contents of the 50 Cc. beaker to boiling, and while boiling add 10 Cc. of solution of Barium Chloride. Stir with a glass rod while boiling, and continue stirring and boiling for about one minute. Set the beaker aside to settle. Then filter through an 11 Cm. ashless filter-paper. Wash out the beaker into the funnel, observing the precautions given on pages 16 to 18. When the beaker is clean wash the filter three times with hot water, or until the last drops running through the funnel give no turbidity when tested on a watch-glass with a drop of Silver Nitrate solution. Place the funnel in an air-bath, and when dry remove the filter-paper and allow the contents to fall on a glazed paper, taking care to detach as much of the precipitate as possible from the filter-paper. Burn the filter-paper as described on page 23, and ignite it in a weighed porcelain crucible. Brush the precipitate from the glazed paper into the crucible and ignite again for about 15 minutes. Allow to cool and add a drop of c. p. strong Sulphuric Acid. Heat *very gently* by moving the burner flame to and fro beneath the crucible. When all the white fumes cease, ignite strongly until the contents of the crucible are white. Remove to a desiccator and weigh when cool. The weight of the Barium Sulphate found multiplied by 12.24 will give the percentage of Sodium Sulphate present in the Soda Ash.

Example:

5 grams Soda Ash taken.

Weight of crucible, cover and $BaSO_4$,	37.2162
Weight of crucible and cover,	36.9337
Weight of $BaSO_4$,	.2825

$.2825 \times 12.24 = 3.46$ per cent. Sodium Sulphate.

DETERMINATION OF SILICA, ALUMINA, FERRIC OXIDE, AND LIME.

Weigh 10 grams of the Ash into a platinum crucible and heat over a blast-lamp until entirely fused. Grasp the crucible with the tongs and dip it into cold water to about one-half its depth. This sudden cooling causes the fused mass to shrink away from the crucible and renders its removal easy with the aid of a glass

rod. Allow the cooled mass to drop into a 50 Cc. porcelain casserole. Pour in enough hot water to cover the button and place a watch-glass on the casserole. Half fill the platinum crucible with c. p. dilute Hydrochloric Acid and pour this into a small beaker. Then wash the crucible into this beaker. Carefully add the contents of the beaker to the casserole, and wash the beaker into the casserole. Place the covered casserole on an air-bath (Fig. 52) and heat gently. When effervescence has ceased take off the cover and wash it with a jet of hot water into the casserole. Add a drop of strong Nitric Acid and allow the contents of the casserole to evaporate to dryness, redissolve in a little dilute Hydrochloric Acid, evaporate again to dryness. Then add about 25 Cc. of dilute Hydrochloric Acid and filter through a 9 Cm. ashless filter-paper. Wash out the casserole with hot water and wash the filter until the last drops from the funnel give no reaction when tested with Silver Nitrate solution. Remove the filter-paper from the funnel while still wet and place it in a weighed platinum crucible. Gently heat with a blast-lamp, and when the paper is dry raise the heat and continue igniting until the contents of the crucible (Silica, SiO_2) are white. Cool the crucible in a desiccator and weigh. Subtract the weight of the crucible, and the difference will be Silica, SiO_2, which multiplied by 10 will give the per cent.

Treat the filtrate from the Silica exactly as described under Silicate Analysis, page 83, and determine the Alumina, Ferric Oxide and Lime in the same manner as described in that method.

DETERMINATION OF MOISTURE.

Weigh a platinum or porcelain dish on the balance pan. Then place a 5-gram weight with the other weights, and in the dish sufficient Soda Ash to balance. Remove the dish to a drying-oven (Fig. 37) and heat at 100° C. for about a half hour. Remove the dish to a desiccator to cool, and then weigh it with its contents. Place again in the drying-oven for another half hour and weigh again. Continue drying and weighing until two successive weights are obtained which do not differ more than two milligrams. The loss of weight multiplied by 20 represents the percentage of Moisture (H_2O) present in the Ash.

Example:

5 grams Ash.

Weight before heating,	37.6700
Weight after first heating,	37.5640
Weight after second heating,	37.5620
Loss of weight,	$.1080 \times 20 = 2.16$ per cent. Moisture.

NOTES OF A COMPLETE SODA ASH ANALYSIS.

Determination of Soda, 3.1 grams taken:

Standard Acid used, 46 Cc., equals 46 per cent. Soda, or 78.66 per cent. Sodium Carbonate.

Determination of Salt, by weight or gravimetrically, 1 gram taken:

Weight of crucible, cover, and AgCl,	35.9873
Weight of crucible and cover,	35.6392
Weight of AgCl,	$.3481 \times 40.85 = 14.22$ per cent. Salt.

Determination of Salt with standard Silver Nitrate or volumetrically, 1 gram taken:

Standard Silver Nitrate used, 24.1 Cc., $\times$ ($.0059 \times 100$) = 14.22 per cent. Salt.

Determination of Salt Cake, 5 grams taken:

Weight of crucible, cover and $BaSO_4$,	37.2162
Weight of crucible and cover,	36.9337
Weight of $BaSO_4$,	$.2825 \times 12.24 = 3.46$ per cent. Salt Cake.

Determination of Silica, Alumina, Ferric Oxide and Lime, 10 grams taken:

Weight of platinum crucible, cover and Silica (SiO_2),	36.9033
Weight of platinum crucible and cover,	36.8605
Weight of Silica (SiO_2),	$.0428 \times 10 = 0.43$ per cent. Silica.

Filtrate divided into two parts and treated as under Silicate Analysis.

Part I., or 5 grams.

Determination of Ferric Oxide, Fe_2O_3:

Weak standard Permanganate, 5.5 Cc., × .0014 = .0077; .0077 × 20 = 0.154 per cent. Fe_2O_3.

Part II., or 5 grams:

Weight of crucible, cover, Alumina and Ferric Oxide,	36.1300
Weight of crucible and cover,	36.0836
Weight of Alumina and Ferric Oxide,	.0464
Weight of Ferric Oxide found from Part I.,	.0077
Weight of Alumina (Al_2O_3),	.0387 × 20 = 0.774 per cent. Alumina.

Filtrates from Parts I. and II. combined to determine Lime.

Parts I. and II., 10 grams.

Strong standard Permanganate, 0.6 Cc., × .005 = .003 × 10 = 0.03 per cent. Lime.

Summary of Analysis:

Soda 46 per cent.	Sodium Carbonate, Na_2CO_3,	78.66
	Salt, or Sodium Chloride, NaCl,	14.22
	Salt Cake, or Sodium Sulphate, Na_2SO_4,	3.46
	Silica, SiO_2,	.43
	Alumina, Al_2O_3,	.77
	Ferric Oxide, Fe_2O_3,	.15
	Lime, CaO,	.03
	Moisture, H_2O,	2.16
		99.88 per cent.

SALT CAKE.

Sodium Sulphate, Na_2SO_4.

Determination of total Sulphate:

Weigh 1 gram of the Salt Cake into a 50 Cc. beaker and add about 10 Cc. of hot water and 5 Cc. of c. p. strong Hydrochloric Acid. Heat gently until dissolved, and filter through a 7 cm. filter paper into a 150 Cc. beaker. Wash out the first beaker on to the filter paper, and wash the filter three times with hot water. Heat the filtrate to boiling, and add 15 Cc. of hot Barium Chloride solution, as described under Soda Ash, determination of Salt Cake, page 56. Stir with a glass rod, filter, dry and ignite the precipitated Barium Sulphate as described on page 56. The weight of $BaSO_4$, multiplied by 61.21, will give the percentage of Sodium Sulphate.

Example, 1 gram taken:

Weight of crucible, cover and $BaSO_4$,	35.9786
Weight of crucible and cover,	34.4950
Weight of $BaSO_4$,	1.4836

$1.4836 \times 61.21 = 90.81$ per cent. Sodium Sulphate.

Determination of Salt, Sodium Chloride, NaCl:

As Salt Cake is made directly from common salt, it is liable to contain more or less of it, according to the care which has been taken in the manufacturing process. Proceed exactly as directed under Soda Ash, pages 54 and 55, using either method.

Determination of Silica, Alumina, Ferric Oxide:

Weigh 10 grams of the Salt Cake, and brush it into a 200 Cc. beaker. Dissolve in about 25 Cc. of dilute Hydrochloric Acid. Filter through a 7 Cm. ashless filter paper into a 500 Cc. porcelain casserole, and wash the beaker on to the filter paper, using a jet of hot water. Wash the filter paper three times with hot water, and place it still wet in a platinum crucible. Heat over a blast lamp until the paper is reduced to ashes. Then place in the crucible about 1 gram of c. p. Sodium Carbonate. Renew the heat and bring the contents of the crucible to fusion. Cover the crucible, and when cool, add, *cautiously*, about 5 Cc. of dilute Hydrochloric Acid. Empty the crucible into the casserole, wash it with a jet of hot water, and wash the cover off also into the casserole. Place the casserole on an air bath and evaporate to

dryness. Then redissolve in a little dilute Hydrochloric Acid, and proceed exactly as described on pages 81 to 84, omitting the determination of Lime.

Determination of Moisture, H_2O:

Proceed exactly as described on page 57, determination of Moisture in Soda Ash.

Example of a complete analysis of Salt Cake:

Sodium Sulphate, Na_2SO_4,	90.81
Sodium Chloride, NaCl,	6.61
Moisture, H_2O,	1.53
Silica, SiO_2,	.50
Alumina, Al_2O_3,	.28
Ferric Oxide, Fe_2O_3,	.12
	99.85 per cent.

Sodium Nitrate, $NaNO_3$.

Determination of Sodium Chloride, NaCl:

Common salt is present in commercial nitrate as an impurity. To determine its percentage, proceed as follows: Weigh 5 grams of the nitrate into a 100 Cc. porcelain dish, and add about 50 Cc. of cold water. Stir with a glass rod until dissolved. Add two drops of Potassium Chromate Indicator, and with a burette, as before described, drop in sufficient Standard Silver Nitrate Solution to produce the proper red color. The number of Cc. of Standard Solution used gives the weight of Sodium Chloride present, and this result, multiplied by 20, gives the percentage.

Example, 5 grams taken:

Standard Silver Nitrate used, 6.1 Cc.

$6.1 \times .0059 \times 20 = 0.72$ per cent. NaCl.

Determination of Moisture, H_2O:

Weigh 10 grams of the Nitrate into a platinum dish, heat for a half hour in the drying oven, as directed on page 57, under Soda Ash. The loss of weight will be moisture.

Example, 10 grams taken:

Weight of dish and Nitrate before heating,	42.2706
Weight after 1st heating,	42.1480
Weight after 2d heating,	42.1350
Weight after 3d heating,	42.1350

Loss of weight, or moisture, 0.1356 grams, or 1.356 per cent. H_2O.

Sodium Bicarbonate, $NaHCO_3$.

The constituents to be determined are Combined Water (H_2O), Carbon Dioxide (CO_2) and Alkali (Na_2O). They are to be calculated to total Bicarbonate.

The impurities are Sodium Monocarbonate (Na_2CO_3), Moisture (H_2O), Sodium Chloride (NaCl) and Sodium Sulphate (Na_2SO_4). The three last named are determined exactly as described under Soda Ash. The Sodium Monocarbonate (Na_2CO_3) is calculated from the determination of the ingredients.

Determination of Water, H_2O:

A piece of platinum foil *a* (Fig. 76), about 3 inches by 1 inch is cut along the dotted lines and bent into the form of a boat as shown in *b*. The boat is placed on the balance pan and weighed. One gram of the bicarbonate is then weighed into it, and the boat is placed in a *hard* glass tube which is arranged as shown in Figure 77 *c*, is a bottle half full of strong Sulphuric Acid to dry the air passing into the tube *d*. This bottle is furnished with two bent glass tubes passing through a rubber stopper; the left hand tube extending below the surface of the acid, the right hand tube passing through another rubber stopper fitted into *d*. *d* is the hard glass tube containing the boat *b*, and connected with the U tube *e* by means of another rubber stopper. The U tube *e* is filled with dry, granular Calcium Chloride, and when not in use the ends are closed by means of short pieces of rubber tubing stoppered by bits of glass rod, as shown in *j j*. It has a loop of platinum wire *k* to hang it to the balance hook when weighing. The tube *e* should be about 4 inches long, and before using, the Calcium Chloride in it must be saturated by Carbonic Acid gas, which is made as described on page 43. The tube *e* is connected with an aspirator bottle *f* of about 2 gallons capacity, having the tablature or outlet *g* furnished with a bent glass tube and a rubber tube closed by the screw pinch cock *h*. The burner *l* is placed under *d*. The apparatus is supported by means of a burette clamp *m*, attached to the stand *n*.

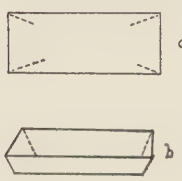


Fig. 76.

Weigh the tube *e* and connect it with *d* and *f*. After placing the boat in the tube *d*, ascertain if the joints are all tight by allowing the water to drop from *g*. Light the burner *l*, and gently heat

the tube *d*. Gradually raise the heat until the tube is red hot, keeping *g* open all the time. Maintain a red heat for about 20 minutes, and then extinguish the burner flame and allow the water to continue running from *g* until the tube *d* is cool. Remove the U-tube *e*, close it with the caps *j*, *j*, hang it on the balance hook by means of the loop *k*, and weigh it. The increase of weight will be the water originally contained in the Bicarbonate.

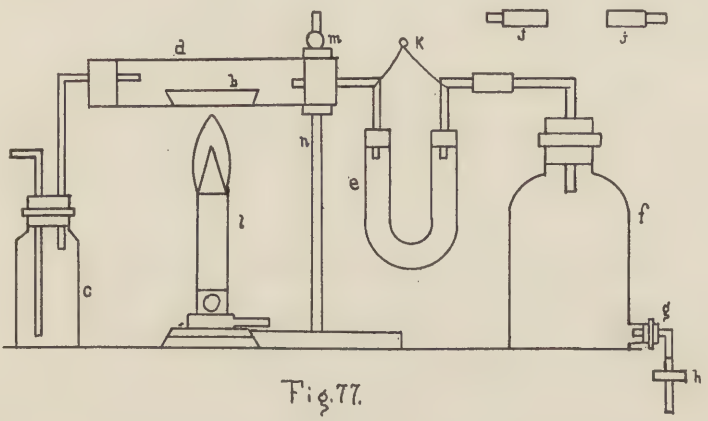


Fig. 77.

Remove the boat *b* and weigh it with its contents; the decrease in weight will be the water plus one-half of the Carbon Dioxide of the Bicarbonate.

Determination of Carbon Dioxide, (CO_2):

Prepare an apparatus according to Figure 78. A 120 Cc. Erlenmeyer flask *a* is supported by the clamp *b* and stand *c*, and is heated by the burner *d*. The flask is closed by a rubber stopper *e*, which has two holes. Through one hole is passed the stem of the separating funnel *f* reaching nearly to the bottom of *a*. A bent glass tube *g* is fitted into the other hole, and connected by a piece of rubber tubing to another glass tube *h*, which goes through a two-hole rubber stopper into an empty 2-oz. glass bottle *j*, reaching nearly to the bottom. Another bent glass tube *k* placed in the other hole, connects by means of rubber tubing, with the bent tube *l* entering the 2-oz. glass bottle *m*, which is nearly one-half full of concentrated Sulphuric Acid. The glass tube *n* connects *m* with a 6-inch U-tube *o* which is filled with pieces of pumice stone saturated with strong Sulphuric Acid. The tube *o* is connected

with another U-tube *p* filled with Anhydrous Copper Sulphate (page 50). The tube *p* is connected by means of a flexible rubber tube *q* with an absorption tube *r* described below. The rubber tube *s* connects *r* with the 8-inch U tube *t* filled with dry, granular

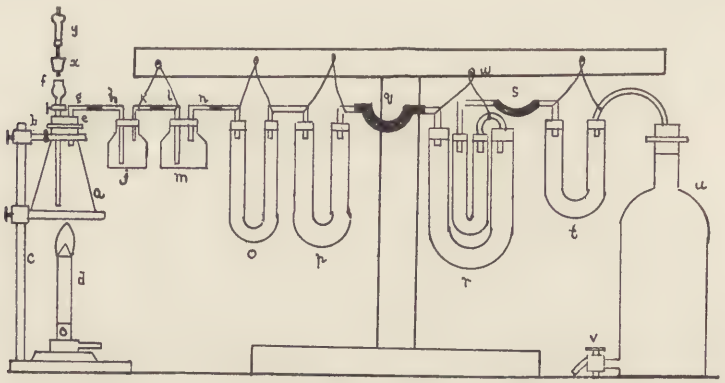


Fig. 78

Calcium Chloride. The 2-gallon aspirator bottle *u* completes the apparatus. It has a glass stop-cock *v* to regulate the flow of water. Instead of an aspirator bottle a filter pump (Figs. 35, 36) may be used.

The absorption tube *r* is composed of a six-inch U-tube, filled with Soda Lime (page 49), and a four-inch U-tube filled with pieces of pumice stone, saturated with strong Sulphuric Acid. It is closed by caps, as described in Figure 77, tube *e*. The pumice stone must first be freed from Chlorides and Fluorides by placing it in a porcelain dish on a sand bath, adding enough strong Sulphuric Acid to cover it, and heating until white fumes appear. When weighed, *r* is hung from the balance hook by means of the loop of platinum wire *w*. Another tube *y*, filled with Soda Lime, is attached to the rubber stopper *x* for the purpose of closing the funnel *f* and purifying the air, which is afterward drawn through the apparatus.

Weigh the absorption apparatus *r* and connect it with the tubes *q* and *s*.

After the apparatus is set up, it is tested for leaks as follows: Insert the stopper *x* in the funnel *f*, and open the funnel stop-cock.

Then open the stop-cock *v* and allow the water to flow freely. A current of air entering at *y* will pass through the whole apparatus. Bubbles of air will be seen in *m*, *o* and *r*. Close the stop-cock in *f*, and if all the joints are tight, the bubbling will stop. If any bubbles are seen, it will show that there is a leak in that part of the apparatus, or in the part to the left of it.

Weigh .5 gram of the Bicarbonate and brush it into the flask *a*. Insert the stopper *e*, connect *g* and *h* and close the stop-cock in *f*. Into *f* pour 25 Cc. of water and insert the stopper *x*. Open the stop-cock *v* about half way and the stop-cock in *f* fully, and permit the water to flow freely into *a*. Close the stop-cock in *f*, take out *x*, and into *f* pour about 20 Cc. of dilute Nitric Acid. Replace *x* and open *v* fully. Open the stop cock in *f* *very cautiously*, and permit the acid to enter *a*. A violent effervescence will ensue, and the liberated Carbon Dioxide (CO_2) will pass through *j*, *m*, *o* and *p* and be absorbed by the Soda Lime in *r*. When the effervescence has subsided, close the stop-cock in *f* and place the lighted burner *d* under *a*. Permit the flame to just touch the bottom of *a*, and gradually heat to boiling. Allow to boil for about two minutes, and extinguish the flame. Open the stop-cock in *f* and partly close *v*, allowing the water to drop rapidly. It will be noticed that the part of the absorption apparatus *r* which contains the Soda Lime has become quite warm. Continue drawing a current of air through until this last-mentioned tube does not feel warm when touched by the hand. Close *v*, remove *r* and insert the caps. Hang it on the balance hook and weigh it. The increase in weight will be the Carbon Dioxide (CO_2) originally contained in the Bicarbonate.

Determination of Alkali, Na_2O:

Weigh 3.1 grams of the Bicarbonate and brush it into a 100 Cc. Erlenmeyer flask. Add about 20 Cc. of cold water and a drop of Methyl Orange Indicator. Remove to a burette and titrate with Standard Acid, as described under Soda Ash. The number of Cc. and tenths used of the Standard Acid will represent the percentage of Alkali (Na_2O) present.

Calculation.

Having determined the constituents and impurities of the Bicarbonate, the next step is to calculate the percentage of actual Sodium

Bicarbonate which is available. The method of calculation is best shown by an example.

Example of Sodium Bicarbonate Analysis:

Determination of Moisture, 10 grams taken:

Weight of platinum dish and Bicarbonate,	45.3983
Weight after first heating,	45.3683
Weight after second heating,	45.3683
Loss of weight,	.0300 or 0.3 per cent. H_2O.

Determination of Sodium Chloride (NaCl), weight taken 2 grams:

Standard Silver Nitrate used, 2 Cc.

$2 \times .0059 = .0118$ (NaCl) $\times 50 = 0.59$ per cent. NaCl.

Determination of Sulphate (Na_2SO_4), 5 grams taken:

Weight of crucible, cover and $BaSO_4$,	34.2900
Weight of crucible and cover,	34.2630
Weight of $BaSO_4$,	.0270

$.0270 \times 12.24 = 0.33$ per cent. Na_2SO_4.

Determination of Water (H_2O):

Weight of boat and Bicarbonate,	2.9920
Weight of boat,	1.9920
Taken for analysis,	1 gram.

Weight of boat and Bicarbonate before heating,	2.9920
Weight after heating,	2.6260
Loss or Water (H_2O) plus one-half Carbon Dioxide (CO_2),	.3660
Weight of Calcium Chloride tube after absorption (*e*, Fig. 77),	30.8530
Weight before absorption,	30.7450
Increase of weight, or H_2O, from Bicarbonate,	.1080
Weight of moisture in 1 gram of Bicarbonate, as found above,	.0030
Combined water of Bicarbonate,	.1050
Loss of weight of boat and Bicarbonate after heating,	0.3660
Water as found above,	0.1080
Difference or one-half of Carbon Dioxide in Bicarbonate,	.2580

$.2580 \times 2 \times 100 = 51.6$ per cent. CO_2 in Bicarbonate.

Determination of Carbon Dioxide (CO_2), weight taken 0.5 gram:

Weight of absorption tube after absorption (*r*, Fig. 78),	131.5092
Weight before absorption,	131.2512
Increase in weight or CO_2 in Bicarbonate,	.2580

$.2580 \times 2 \times 100 = 51.6$ per cent. CO_2 in Bicarbonate.

Determination of Alkali $(Na_2O)_2$, weight 3.1 grams:

Standard Acid used, 36.6 Cc.

Alkali (Na_2O), 36.6 per cent.

Summary.

Sodium Chloride, NaCl,	.59
Sodium Sulphate, Na_2SO_4,	.33
Moisture, H_2O,	.30
Combined Water, H_2O,	10.50
Carbon Dioxide, CO_2,	51.60
Alkali, Na_2O,	36.60

Calculation.

First calculate the percentage of Carbon Dioxide due to the Alkali found.

Alkali (Na_2O) : Carbon Dioxide (CO_2) : : 36.6 : per cent. CO_2 or, supplying the molecular weights:

62 : 44 : : 36.6 : per cent. CO_2

$44 \times 36.6 \div 62 =$ per cent. CO_2.

CO_2 due to $Na_2O = 25.97$ per cent.

This result may also be obtained by multiplying the Alkali percentage by 44 and dividing the result by 62.

Subtract the percentage of CO_2 due to Alkali from the total CO_2, as found above.

Total CO_2, 51.6, minus CO_2 due to Alkali, 25.97, equals 25.63 per cent. Multiply this by 2, to get the CO_2 due to Bicarbonate.

$25.63 \times 2 = 51.26$ per cent.

Calculate this to Bicarbonate:

Carbon Dioxide (CO_2) : Sodium Bicarbonate ($NaHCO_3$) : : 51.26 : per cent. $NaHCO_3$, or, substituting the molecular weights:

44 : 84 : : 51.26 : per cent. $NaHCO_3$.

$84 \times 51.26 \div 44 =$ per cent. $NaHCO_3$, or 97.90 per cent.

This result may also be obtained by multiplying the percentage of CO_2 due to $NaHCO_3$ by 84, and dividing the result by 44.

Calculate the CO_2 due to Bicarbonate into terms of Alkali (Na_2O) due to Bicarbonate on the basis that two molecules Carbon Dioxide (or 2 CO_2) are equivalent to one of Alkali (Na_2O), that is, 2 CO_2 = Na_2O, we have the proportion :

2 CO_2 : Na_2O : : 51.26 : per cent. Alkali due to Bicarbonate, or supplying the molecular weights:

88 : 62 : : 51.26 : per cent. Alkali due to Bicarbonate.

$62 \times 51.26 \div 88 = 36.11$ per cent. Na_2O due to Bicarbonate.

This result is also obtained by multiplying the percentage CO_2 due to Bicarbonate by 62 and dividing by 88.

Subtract this from the total Alkali found, 36.6 minus 36.11 equals 0.49 per cent. Alkali (Na_2O) due to Monocarbonate (Na_2CO_3).

Calculate this into Monocarbonate (Na_2CO_3):

Na_2O : Na_2CO_3 : : 0.49 : per cent. Na_2CO_3.

Supplying molecular weights, 62 : 106 : : 0.49 : per cent. Na_2CO_3

$106 \times 0.49 \div 62 = 0.84$ per cent. Na_2CO_3.

Results of Analysis.

Sodium Bicarbonate, $NaHCO_3$,	97.90
Sodium Carbonate, Na_2CO_3,	.84
Sodium Chloride, NaCl,	.59
Sodium Sulphate, Na_2SO_4,	.33
Moisture, H_2O,	.30
	99.96 per cent.

Condensed Rules for Calculations.

1. Multiply the Alkali (Na_2O) percentage by 44, and divide the result by 62. This gives the Carbon Dioxide due to total Alkali.

2. Subtract this result from the total Carbon Dioxide, and multiply the remainder by 2. This gives the Carbon Dioxide due to Bicarbonate.

3. Multiply this result by 84 and divide by 44. This gives the percentage of Sodium Bicarbonate ($NaHCO_3$).

4. Multiply the Carbon Dioxide percentage found by Rule 2, by 62 and divide by 88. Subtract the result from the percentage of total Alkali (Na_2O). This gives the percentage of Alkali due to Monocarbonate.

5. Multiply this result by 106 and divide by 62. This gives the percentage of Monocarbonate (Na_2CO_3).

Potash, Potassium Carbonate K_2CO_3.

Determination of Alkali (K_2O), and total Potash (K_2CO_3).

Weigh 2 grams of Potash, and brush it into a 120 Cc. Erlenmeyer flask. Add 25 Cc. of cold water and allow to dissolve. Add one drop of Methyl Orange Indicator, remove to a burette and titrate with Standard Acid as described under Soda Ash (page 53). The number of Cc. and tenths of the Acid used, multiplied by .047, and this result multiplied by 50 will be the percentage of Alkali (K_2O). The Alkali percentage multiplied by 138 and divided by 94, will give the percentage of total Potash (K_2CO_3).

Example, 2 grams taken:

Standard Acid used 24.3 Cc.

$24.3 \times .047 \times 50 = 57.1$ per cent. Alkali, K_2O.

$57.1 \times 138 \div 94 = 83.82$ per cent. Potash, K_2CO_3.

Determination of Water, H_2O:

Weigh 10 grams into a weighed platinum crucible and place over a Bunsen burner. Allow the flame to just touch the bottom, keeping it red hot for about 15 minutes. Place in a desiccator, and when cool weigh it. Heat again for about 10 minutes, and repeat the weighing. When two weights are obtained which do not differ more than 10 milligrams, consider the loss as Water (H_2O).

Example, 10 grams taken:

Weight of crucible, cover, and Potash before heating,	66.578
Weight after first heating.	65.198
Weight after second heating,	64.918
Loss or Water (H_2O),	1.660 grams.

$1.660 \times 10 = 16.6$ per cent. Water (H_2O).

Determination of Chloride (KCl):

Weigh 10 grams, and proceed exactly as described under Soda Ash, determination of Salt (page 55). The number of Cc. and tenths of the Standard Silver Nitrate used, multiplied by .0075, and this result multiplied by 10 will give the percentage of Potassium Chloride (KCl) present in the Potash.

Example, 10 grams taken:

Standard Silver Nitrate, 0.5 Cc.

$0.5 \times .0075 \times 10 = 0.37$ per cent. Chloride (KCl).

Potassium Nitrate, Saltpeter, KNO_3.

Determination of Water (H_2O):

Proceed as directed under Sodium Nitrate, page 61.

Determination of Chloride (KCl):

Proceed as directed under Sodium Nitrate, page 61, multiplying the Standard Silver Nitrate by .0075, and multiply this result by 20 to get the percentage of Chloride (KCl).

LIME.

The various kinds of lime used in glass manufacture are: Shell lime, made by burning oyster shells, and water-slaking the quick lime obtained; Stone lime, made by crushing limestone, calcite or marble, and containing, theoretically, 100 per cent. Calcium Carbonate ($CaCO_3$); Dolomite lime, made by burning dolomite. This last-mentioned variety may contain upward of 40 per cent. Magnesia (MgO). Ohio lime is of this kind.

Determination of Lime (CaO) and Magnesia (MgO):

Weigh 1 gram and brush it into a 150 Cc. beaker. Cover the beaker with a watch glass and add about 50 Cc. of cold water, and then 20 Cc. of concentrated Hydrochloric Acid—adding the acid carefully. When effervescence has ceased, wash off the watch glass into the beaker and carefully boil until dissolved. Add a drop of concentrated Nitric Acid and boil again. Add sufficient strong Ammonia to give a decided odor, boil and filter through a 7 Cm. filter paper into a 200 Cc. graduated flask. Wash three times with hot water, filling the funnel each time. Cool the flask by allowing hydrant water to run over the outside, and when cool, fill to the mark with cold distilled water. Mix well by shaking the flask. With a pipette, take out 50 Cc. and allow it to run into a 200 Cc. beaker. Add a little strong ammonia and heat the contents of the beaker to boiling, and add about 30 Cc. of hot solution of Ammonium Oxalate. Stir and boil for two minutes, and set aside to allow the precipitated Calcium Oxalate to settle. When the precipitate has settled clear, filter through an 11 Cm. ashless filter-paper into a 300 Cc. beaker, and wash the first beaker with a jet of hot water making sure to remove all the precipitate to the filter-paper, and using the "policeman" as described on page 17. Wash the filter with hot water until the last drops running through show no turbidity when tested with Silver Nitrate.

Take the filter out of the funnel and place it in a 500 Cc. flask (Fig. 12), and add about 200 Cc. hot water. *Carefully* pour about 15 Cc. of concentrated Sulphuric Acid into the flask. This is performed by adding about 1 Cc. at a time, and shaking the flask at each addition. Remove the flask to a burette and titrate with Strong Standard Permanganate. Add this carefully until *one drop* gives a decided pink color, which remains for at least five minutes on shaking.

The number of Cc. and tenths of Permanganate used, multiplied by .005, will be the amount of Calcium Oxide (CaO) or Lime present. As 50 Cc. of the solution was taken and this represents 0.25 grams in weight, we multiply the above result by 400 to get the percentage.

The precipitate from the Calcium Oxalate contained in the 300 Cc. beaker is allowed to cool. Add sufficient strong Ammonia to give a decided odor, and about 30 Cc. of solution of Sodium Phosphate and set aside over night. Filter through a 9 Cm. ashless filter-paper, and wash out the beaker with dilute Ammonia (1 to 3 of water). Carefully remove all the precipitate from the beaker and stirring rod, using a policeman. This must be carefully performed, as the precipitate adheres very strongly to the sides of the beaker.

Wash the filter four times with dilute Ammonia and place the funnel in a drying oven. When dry, remove from the funnel, and allow the precipitate to fall on a piece of glazed paper. Burn the filter-paper on a platinum wire (page 23), and ignite the charred mass in a weighed porcelain crucible. When reduced to ashes allow the crucible to cool and add a drop of strong Nitric Acid. Carefully evaporate with the burner flame and heat until white. Allow the crucible to become cool again, and brush into it the precipitate from the glazed paper. Ignite strongly, cool again, and add a couple of drops of strong Nitric Acid. Evaporate carefully to avoid spattering, and ignite until the contents of the crucible are white. Remove to a desiccator and weigh when cool. The weight of the Magnesium Pyro-phosphate ($Mg_2P_2O_7$) found, multiplied by .36 gives the Magnesia (MgO) in 0.25 grams of Lime taken for analysis, and this weight multiplied by 400 gives the percentage of Magnesia (MgO).

Example I, 100 Cc. of Lime Solution taken equivalent to 0.5 grams Toledo Lime:

Strong Standard Permanganate, 28.7 Cc.

$28.7 \times .005 \times 400 = 57.4$ per cent. Lime (CaO).

Weight of crucible, cover, and $Mg_2P_2O_7$,	28.1050
Weight of crucible and cover,	27.8330
Weight of $Mg_2P_2O_7$,	.2720

$0.272 \times .36 \times 400 = 39.168$ per cent. Magnesia (MgO).

Example II, Burnt Limestone:

Strong Standard Permanganate 40.3 Cc.

$40.3 \times .005 \times 400 = 80.6$ per cent. Lime (CaO).

Weight of crucible, cover and $Mg_2P_2O_7$,	27.8254
Weight of crucible and cover,	27.8010
Weight of $Mg_2P_2O_7$,	.0244

$0.0244 \times .36 \times 400 = 3.52$ per cent. Magnesia (MgO).

Determination of Carbon Dioxide (CO_2):

The apparatus shown in Figure 78 is used in this determination. For a ground Rock-Lime take 0.5 grams, and for a burnt Lime 1 gram. Weigh out the Lime, brush it into the flask *a*. Weigh the absorption tube *r* and connect it with *q* and *s*. Make all the necessary connections and proceed exactly as described under Sodium Bicarbonate on pages 63 to 65. The increase in weight of the tube *r* will be Carbon Dioxide. To get the percentage multiply by 200 in case of a Limestone, and by 100 in case of a burned Lime.

Example, Burned Lime 1 gram taken:

Weight of absorption tube after,	66.1383
Weight of absorption tube before,	66.1030
Weight of Carbon Dioxide (CO_2),	.0353

$.0353 \times 100 = 3.53$ per cent. Carbon Dioxide.

Determination of Water, H_2O:

Weigh 1 gram of the Lime into a weighed platinum crucible. Ignite over a blast lamp until the weight is constant, that is, until the last two weighings do not differ more than 5 milligrams. The

loss will be Water and Carbon Dioxide. Subtract from this the Carbon Dioxide as found by the foregoing method. The difference will be Water (H_2O).

Example. Burned Lime, 1 gram taken:

Weight of crucible, cover and Lime,	25.2244
Weight after first heating,	25.1684
Weight after second heating,	25.1009
Weight after third heating,	25.1004
Loss of weight, or Water, plus Carbon Dioxide,	.1240
Weight of Carbon Dioxide as found before,	.0353
Weight of water,	.0887

$.0887 \times 100 = 8.87$ per cent. Water (H_2O).

Determination of Silica, Alumina and Ferric Oxide:

Weigh 1 gram of the Lime and brush it into a 100 Cc. beaker. Cover with a watch glass and proceed to dissolve in dilute Hydrochloric Acid. When dissolved, filter through a 7 Cm. ashless filter-paper into a 200 Cc. porcelain casserole. Wash the beaker onto the filter, and wash the latter three times with hot water. Place the casserole on an air-bath to evaporate. Remove the wet filter-paper from the funnel and place it in a platinum crucible. Ignite over a blast lamp until the paper is reduced to ashes. Then place in the crucible about 1 gram of dry c. p. Sodium Carbonate, and heat until fused. When cool, cover the crucible, add a little water and a few drops of concentrated c. p. Hydrochloric Acid. Keep the cover on while effervescence is noticed and add more acid, a few drops at a time, until the fused mass is dissolved. Pour the contents of the crucible into the casserole, wash the cover and crucible with a jet of hot water, allowing the washings to fall into the casserole. To the solution add a drop of concentrated Nitric Acid and evaporate to dryness. Allow to cool, and add just sufficient dilute Hydrochloric Acid to redissolve. Evaporate again to dryness and redissolve again in dilute Hydrochloric Acid. Filter through a 11 Cm. ashless filter-paper and proceed exactly as described under Typical Silicate Analysis, on pages 83 and 84, paragraphs 2, 3, and 4.

Example of Analysis of Burned Lime:

Lime, CaO,	80.60
Magnesia, MgO,	3.52
Carbon Dioxide, CO_2,	3.53
Water, H_2O,	8.87
Alumina, Al_2O_3,	1.23
Ferric Oxide, Fe_2O_3,	.20
Silica, SiO_2,	1.77
	99.72 per cent.

MANGANESE.

Ores of Manganese are valued according to their oxidizing power, and this is expressed in terms of the Peroxide, or Dioxide (MnO_2). To determine the oxidizing power, proceed as follows:

Prepare an apparatus, as shown in Fig. 79. The ring stand *a* supports the 500 Cc. flask *b*, which is fitted with a rubber stopper *c*, having one hole. The bent tube *d* passes through this hole and dips just below the surface of the water contained in the 50 Cc. beaker *e*. Pour into the flask about 90 Cc. of c. p. dilute Sulphuric Acid and drop in one gram of piano wire which has been carefully weighed. Insert the stopper and with the burner *f* gently heat until the wire is dissolved. Remove the stopper and add 0.5 gram of the finely-powdered sample of Manganese ore. It is well to have this in a small, wide, glass tube and to drop the tube directly into the flask. Replace the stopper and boil gently until the sample is dissolved. Place the end of the tube *d* into the water in *e* and allow the water to recede through *d* into the flask. The water in *e* should have been previously boiled to expel any air it may contain. Add a little more boiled water to *e* and then add about 200 Cc. of boiled water to the contents of the

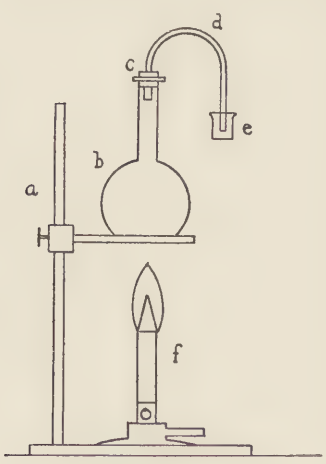


Fig. 79.

flask *a*, allowing it to wash down the inner surface of the flask. Remove the flask to a burette and titrate with strong Standard Permanganate. The number of Cc. and tenths used, multiplied by .01 will give the amount of iron which remains unoxidized by the Manganese ore, and this deducted from the total Iron (1 gram) leaves the Iron which has been oxidized. This result, multiplied by 87 and divided by 112, will give the amount of Peroxide (MnO_2) present in the ore, which multiplied by 200 gives the percentage. The percentage of Peroxide, multiplied by 16 and divided by 87, gives the percentage of available Oxygen.

Example, 0.5 gram taken:

Iron wire,	1.0000 gram.
Strong Standard Permanganate, 47.8 Cc. × .01 =	.4780
Difference, or Iron oxidized by the Manganese ore,	.5220

$.5220 \times \frac{87}{112} = .4055 \times 200 = 81.10$ per cent. MnO_2.

$81.1 \times \frac{16}{87} = 14.85$ per cent. Available Oxygen.

Test for Cobalt in Manganese Ore.

Some Manganese ores contain Cobalt and should be rejected, as even a small percentage of this impurity will give a bluish tint to flint glass. To test for Cobalt, dissolve about 1 gram in Hydrochloric Acid, add a drop of Nitric Acid and boil. Dilute with an equal volume of water, and make alkaline with Ammonia. Add an excess of Ammonium Sulphide and filter. Heat a platinum wire, with the end bent into a small loop, red hot in a burner flame and dip it in some powdered borax. Heat again, and when the borax is in a glassy bead, touch it to the filter. Place in the flame and fuse the bead. A deep blue color indicates Cobalt.

Arsenic or Arsenious Oxide, As_2O_3.

Weigh 1 gram and brush it into a 500 Cc. graduated stoppered flask. Add about 10 Cc. of Aqua Regia (page 43), and when dissolved fill the flask to the mark with cold water. Insert the stopper and mix well by shaking. With a graduated pipette remove 100 Cc. (0.2 grams) and allow it to run into a 200 Cc. beaker. To this add strong Ammonia until a decided odor is produced. Then add about 25 Cc. of Magnesia Mixture (page 49). Stir well and allow to stand for 24 hours. Filter through a 9 Cm.

filter-paper, and with a jet of dilute Ammonia (1 in 4) wash all the precipitate on to the filter-paper. The Ammonia used should have a few drops of Nitric Acid added to it. Be sure to get all the precipitate out of the beaker by using a "policeman." Wash about three times with the Ammonia. When well washed place a *weighed* porcelain crucible under the funnel, and stir the precipitate with a fine jet of dilute Nitric Acid (1 in 2). When the precipitate is dissolved, wash twice with a jet of hot water. Place the crucible on a sand bath and evaporate to dryness. When dry, ignite over a Bunsen burner, gently at first, and then strongly. Remove the crucible and cover to a desiccator, and weigh when cool. The weight of the contents of the crucible, Magnesium Pyro-Arsenate ($Mg_2As_2O_7$) multiplied by 197, and divided by 309 gives the weight of Arsenious Oxide (As_2O_3), and this multiplied by 500 will give the percentage.

Example, 0.2 grams taken:

Weight of crucible, cover, and $Mg_2As_2O_7$,	37.2003
Weight of crucible and cover,	36.8882
Weight of $Mg_2As_2O_7$,	.3121

$.3121 \times \frac{197}{309} \times 500 = 99.45$ per cent. As_2O_3.

LEAD.

Litharge, PbO. Red Lead, Pb_3O_4.

Dissolve 0.5 grams in a small beaker in the least possible amount of concentrated Nitric Acid, and add an equal volume of hot water. Filter through a 9 Cm. filter-paper and allow the filtrate to run into a *weighed* porcelain crucible. Wash with a jet of hot water until the washings running through the funnel are neutral when tested with Litmus paper. Place the crucible on a sand bath, add about 1 Cc. of c. p. concentrated Sulphuric Acid and evaporate to dryness. When dry, ignite over a Bunsen burner until the contents of the crucible are white. Remove to a desiccator and weigh when cool. If the sample is Litharge multiply the Lead Sulphate found by 221 and divide by 301, this gives the weight of Litharge (PbO). This weight multiplied by 200 gives the percentage. If the sample is Red Lead multiply the Lead Sulphate found by 0.753. This result multiplied by 200 gives the percentage of Red Lead (Pb_3O_4).

Example, Litharge 0.5 gram taken:

Weight of crucible, cover, and $PbSO_4$,	15.5371
Weight of crucible and cover,	14.8580
Weight of Lead Sulphate, $PbSO_4$,	.6791

$.6791 \times \frac{223}{303} \times 200 = 99.72$ per cent. PbO.

Barium Carbonate, Baryta, $BaCO_3$.

Determination of Total Carbonate ($BaCO_3$).

Weigh out 1 gram and brush it into the flask *a* Fig. 78. Weigh the absorption tube *r*, and make all necessary connections. Proceed exactly as described on page 65 under Determination of Carbon Dioxide in Sodium Bicarbonate. The increase in the weight of *r*, multiplied by 196 and divided by 48, gives the weight of Barium Carbonate ($BaCO_3$). This result multiplied by 100 gives the percentage.

Another Method.

Based on the precipitation as Barium Sulphate ($BaSO_4$), and calculation of the equivalent Barium Carbonate ($BaCO_3$).

Weigh 0.5 grams of the sample, and proceed exactly as described under Determination of Salt Cake in Sodium Carbonate (page 55). The weight of the Barium Sulphate found multiplied by 196 and divided by 232 gives the weight of Barium Carbonate ($BaCO_3$), and this result multiplied by 200 gives the percentage.

Zinc Oxide. ZnO.

Determination of Total Oxide:

Weigh 0.5 gram and dissolve it in a 250 Cc. beaker in concentrated Hydrochloric Acid. Add a couple of drops of concentrated Nitric Acid and heat to boiling. Dilute with an equal quantity of water, add enough strong Ammonia to give a decided odor and boil for a few minutes. This will precipitate any Iron that may be present. Filter through a 9 Cm. filter-paper into a 750 Cc. beaker and wash the filtrate with hot water until the last few drops running through show no reaction when tested with Silver Nitrate. To the filtrate add enough Hydrochloric Acid to give a strong reaction when tested with Litmus paper.

Remove the beaker to a burette and titrate with Standard Potassium Ferrocyanide. Add the Standard Solution cautiously, and

toward the end of the test take out a drop from the beaker with a glass rod and place it in contact with a drop of Uranium Nitrate Indicator, which is on a china plate. A red color shows that the test is finished. It will be noticed in adding the Standard Solution that the precipitate which forms at first is coarse, but on further addition of the Standard Solution it becomes fine. When this stage is reached, it is time to begin taking out drops to be tested with the indicator.

The number of Cc. and tenths of the Standard Ferrocyanide, multiplied by the strength as found per page 39, gives the weight of Zinc Oxide (ZnO) present, and this result, multiplied by 200, will give the percentage.

Example, 0.5 grams taken. Standard Ferrocyanide 1 Cc. = 0.1246 gms. Zn:

Standard Potassium Ferrocyanide, 39.6 Cc.

$39.6 \times .01246 \times 200 = 98.68$ per cent. ZnO.

Determination of Moisture, H_2O:

Weigh 1 gram into a weighed platinum dish, and heat in a drying oven to constant weight, proceeding exactly as described on page 57, in Determination of Moisture in Soda Ash. The loss of weight, multiplied by 100, gives the percentage of Moisture (H_2O).

Example, 1 gram taken:

Weight of whole first,	35.4996
Weight after drying,	35.4866
Loss of weight,	.0130

Moisture, 1.3 per cent.

FLUORIDES.

The Fluorides used in glass-making are two, namely: Fluorspar or Calcium Fluoride (CaF_2), and Cryolite. The former is a compound of the gas Fluorine (F), with the metal or base Calcium (Ca). The latter is a compound of Fluorine, with the metals or bases Sodium (Na) and Aluminium (Al). Owing to the difficulty experienced in properly estimating the proportion of Fluorine in Fluorides, the methods given below for both minerals can be regarded only as approximate.

Fluorspar, Calcium Fluoride, CaF_2.

Weigh 1 gram into a weighed platinum dish and place on a sand-bath. Add, *very cautiously*, drop by drop, concentrated c. p. Sulphuric Acid until the mineral is decomposed. Heat on the sand-bath until dry, raising the heat to a dull red until no more white fumes are given off. Remove the dish to a dessicator, and when cool weigh it with contents. The Sulphuric Acid replaces the Fluorine of the Fluoride, forming Calcium Sulphate ($CaSO_4$). The weight of the Calcium Sulphate found, multiplied by 78 and divided by 136, gives the weight of Calcium Fluoride (CaF_2) present, and this multiplied by 100 gives the percentage.

Example, 1 gram taken:

Weight of dish and $CaSO_4$,	59.3370
Weight of dish,	57.6072
Weight of $CaSO_4$,	1.7298

$1.7298 \times \frac{78}{136} \times 100 = 99.21$ per cent. CaF_2.

(When treating a Fluoride with Sulphuric Acid, care should be taken to perform the operation in a place where there is a strong current of air, preferably an upward draught, as the liberated Fluorine is dangerous to breathe, and will attack any glass objects in the vicinity.)

Cryolite.

Determination of Fluorine (F):

Weigh 1 gram and mix it in a weighed platinum crucible with 10 grams of c. p. Calcium Carbonate ($CaCO_3$). Heat for one hour over a Bunsen burner, keeping the bottom of the crucible a dull red. Allow to cool and add hot water; as the mass crumbles wash it into a platinum dish and heat on a sand-bath until it appears decomposed (about a half hour). Filter through a 11 Cm. filter-paper with gentle suction and wash thoroughly with hot water. When all the precipitate has been removed from the dish, place the latter beneath the funnel. Cover the funnel with a watch glass, and cautiously add dilute Acetic Acid (1 in 2), moving the watch glass a little to one side while adding the acid. When the precipitate is all dissolved, wash off the watch glass with hot water and wash the filter-paper thoroughly. Remove the dish to a sand-bath and evaporate to dryness, and continue heating until no more odor

of Acetic Acid can be perceived. Add a little hot water and filter on a 9 Cm. ashless filter-paper. Wash thoroughly with hot water. Place the wet filter-paper and contents in a weighed platinum crucible, heat gently with a Bunsen burner until the paper is charred, and then strongly until the contents of the crucible are white. Cool in a dessicator and weigh. The weight of the contents of the crucible or Calcium Fluoride, multiplied by 38 and divided by 78, gives the weight of Fluorine (F) in the Cryolite.

Example, 1 gram taken:

Weight of crucible and CaF_2,	57.9140
Weight of crucible,	56.7938
Weight of CaF_2,	1.1202

$1.1202 \times \frac{3}{7}\frac{8}{8} = .5457 \times 100 = 54.57$ per cent. Fluorine.

If desired, the above result may be checked by adding c. p. concentrated Sulphuric Acid to the Calcium Fluoride in the crucible, evaporating to dryness and igniting, as described in Fluorspar. The Calcium Sulphate thus found, multiplied by 38 and divided by 136, gives the amount of Fluorine which should agree with the above just found.

Determination of Sodium (Na):

Weigh 1 gram and proceed exactly as described in paragraph 9 Silicate Analysis on page 85. The Chlorides found are to be considered as Sodium Chloride (NaCl) which when multiplied by 23 and divided by 58 gives the weight of Sodium (Na), this multiplied by 100 gives the percentage.

Example, 1 gram taken:

Weight of dish and NaCl,	33.4290
Weight of dish,	32.6197
Weight of NaCl,	.8093

$.8093 \times \frac{2}{5}\frac{3}{8} = .3209 \times 100 = 32.09$ per cent. Na.

Determination of Aluminium, Al.

Weigh 1 gram, mix it with 5 grams of the Silicate Flux (page 46). Fuse in a platinum crucible. Proceed as described in paragraphs 2 and 4 of Silicate Analysis, pages 83 and 84. The precipitate obtained by precipitation with Ammonia in paragraph 4 is ignited and weighed as Alumina (Al_2O_3), which when multiplied by 54, and divided by 102, gives the weight of Aluminium (Al).

Example, 1 gram taken:

Weight of crucible, cover, and Al_2O_3,	37.1020
Weight of crucible and cover,	36.8744
Weight of Al_2O_3,	.2276

$.2276 \times \frac{54}{102} = .1204$; $.1204 \times 100 = 12.04$ per cent. Aluminium.

Summary of Cryolite Analysis:

Fluorine found by Acetic Acid treatment,	54.57 per cent.
Sodium found by $CaCO_3$ and NH_4Cl treatment,	32.09 per cent.
Aluminium found by fusion with Silicate Flux,	12.04 per cent.
	98.70 per cent.

SILICATES.

The compounds of Silica (SiO_2) which are considered here are: Clay, Fire Brick, Feldspar, Sand and Glass. As similar methods are used in the determination of the constituents of all of the above, a scheme is given for a typical Silicate Analysis, which with variations will serve as a method of analysis for any of the above.

Typical Silicate Analysis.

Determination of Silica (SiO_2), Alumina (Al_2O_3), Ferric Oxide (Fe_2O_3), Lime (CaO), and Magnesia (MgO):

1. Weigh 1 gram of the pulverized sample and mix it thoroughly with 5 grams of Silicate flux. Place the mixture in a platinum crucible, and by means of a Bunsen burner, heat until all is in a state of quiet fusion and no more bubbles are seen to escape. Grasp the crucible with a pair of tongs (Fig. 38) and dip it in cold water, allowing the water to reach about one-half the height of the crucible and taking care that no water enters. This will cause the fused mass to suddenly cool and shrink away from the crucible, thus loosening it and making it easy to remove. Loosen the mass with the aid of a glass rod and allow it to drop into a 300 Cc. casserole. Add about 100 Cc. of hot water and set the casserole aside. Pour into the crucible about 10 Cc. of dilute Hydrochloric Acid and when the remainder of the fused mass is dissolved, empty the crucible into a small beaker. With a jet of hot water wash the crucible out into this beaker. Cover the casserole with a watch glass and carefully pour into it the solution from the small beaker. taking care that nothing is lost by effervescence. Wash the beaker

into the casserole and add to the contents of the latter about 20 Cc. of concentrated Hydrochloric Acid. Place the casserole on a water-bath, or on an air-bath, and heat until all is dissolved. If the sample has been thoroughly pulverized, and the fusion properly conducted, the fused mass will completely dissolve and no flocculent particles will be seen floating about, and also there will be no gritty material in the bottom of the casserole. Remove the watch glass, wash it into the casserole with a jet of hot water and evaporate the solution to dryness. As it becomes dry, stir it and turn it over with a glass rod, the end of which has been flattened into the shape of a spatula (Fig. 80). When nearly dry, remove from the bath and grind up with a glass pestle. Replace on the bath and allow to come to dryness. When dry, remove again from the bath and grind thoroughly with the pestle. Cover the casserole with a watch glass and place it in a drying oven. Heat with a Bunsen burner and regulate the heat so that the thermometer stands at 120° C., never allowing it to go above 150° C. When the casserole has been in the oven for about half an hour, remove it and allow it to cool. Grind up the contents again and observe if any acid odor is given off. If so, replace in the oven for another half hour. If not, add about 50 Cc. of dilute Hydrochloric Acid, stir with the flattened rod and wash off the rod and pestle with a jet of hot water. Place again on the water-bath or air-bath and heat until all the contents except the Silica is dissolved. Stir with an ordinary glass rod while heating. Filter through a 9 Cm. ashless filter-paper into a 200 Cc. graduated flask. When the casserole has been emptied on to the filter-paper, remove the flask, set it aside and place a 500 Cc. beaker beneath the funnel. Wash the casserole with a jet of hot water, as shown in Figure 25, and remove all the Silica to the filter. Wash off the glass rod, and with a jet of hot water directed around the filter paper, thoroughly wash the Silica. Continue washing until the last drops running through the funnel show no turbidity when tested with Silver Nitrate Solution. Place the wet paper in a *weighed* platinum crucible, heat over a Bunsen burner, and when the paper is charred, raise the heat and keep the crucible red hot until the Silica is white. Cool in a dessicator and weigh. The weight of the Silica (SiO_2) found, multiplied by 100, gives the percentage.

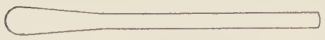


Fig. 80.

2. Filtrate from the Silica:

This contains the Alumina (Al_2O_3), Ferric Oxide (Fe_2O_3), Lime (CaO), and Magnesia (MgO). The contents of the 500 Cc. beaker are evaporated to a small bulk, and poured into the 200 Cc. flask. The beaker is washed with a jet of cold water into the flask. Then place the flask under a hydrant and allow the water to run over the outside to cool it to 15° C. Fill it to the mark with cold water, insert the stopper and mix by shaking. With a graduated pipette take out 100 Cc. and allow it to run into a 150 Cc. beaker. Pour the rest of the liquid into another 150 Cc. beaker, and wash *both* the flask and pipette into this last beaker. To each beaker add a drop of Nitric Acid, and heat to boiling. Remove from the heat, and to each add sufficient strong Ammonia to give a decided odor. Boil again for a few minutes, and set the beakers aside for the precipitates to settle. Select ashless filter-papers large enough to take the precipitate, and if necessary use a filter pump (Fig. 35), with the filtering flask and platinum cone (Figs. 30, 31). Filter the contents of both beakers into 200 Cc. beakers, and when all is poured in, substitute two other beakers (250 Cc.) to receive the washings. Wash thoroughly with hot water, and test the last drops running through the funnels with Silver Nitrate. The washings contained in the 250 Cc. beakers are to be boiled down to a small bulk and added to their respective 200 Cc. beakers containing the filtrates.

3. Determination of Ferric Oxide (Fe_2O_3):

Place a 100 Cc. Erlenmeyer flask under one of the funnels, and pour on the filter-paper about 25 Cc. of dilute c. p. Sulphuric Acid (1 in 5). This will dissolve the precipitate on the filter-paper. Wash three times with hot water. Place in the flask a piece of platinum foil in contact with a piece of c. p. stick Zinc. Allow the flask to stand about one hour and then empty it into a 200 Cc. beaker, keeping the Zinc and platinum foil in the flask. Wash out the flask with a little cold water, and pour the washings into the beaker. Place the beaker under a burette and titrate with Weak Standard Permanganate. The number of Cc. and tenths used, multiplied by .0014 gives the Ferric Oxide, this is to be multiplied by 2, because we divided our previous solution (paragraph 1) into two parts. The number of Cc. and tenths multiplied by .00128, and this result multiplied by 200 gives the per-

centage of *Ferrous Oxide* (FeO), in case the Iron is present in that form.

4. Determination of Alumina (Al_2O_3):

Place the filter-paper and contents from the other funnel in a weighed platinum crucible, burn off the paper, and ignite. Cool in a desiccator and weigh. The contents of the crucible will be Alumina and Ferric Oxide. Subtract the Ferric Oxide as found in paragraph 3, and multiply the result by 200. This will give the percentage of Alumina (Al_2O_3).

5. Determination of Lime (CaO):

We have two 200 Cc. beakers to which have been added the washings from the Alumina and Ferric Oxide precipitates (paragraphs 3 and 4). Pour them into a larger beaker and wash the 200 Cc. beakers into it. Add enough strong Ammonia to give a decided odor, boil and add about 15 Cc. of hot Ammonium Oxalate solution. Proceed as directed under Lime Analysis (page 70). The number of Cc. and tenths of Strong Standard Permanganate used multiplied by .005, and this result multiplied by 100 gives the percentage of Lime (CaO).

6. Determination of Magnesia (MgO):

To the filtrate from the Calcium Oxalate precipitate, obtained in paragraph 5, add 1 Cc. of strong Nitric Acid, and evaporate to about 150 Cc. Then add enough strong Ammonia to give a decided odor, and about 10 Cc. of Sodium Phosphate. Set aside over night, and proceed as directed in Lime Analysis (page 71). The $Mg_2P_2O_7$ found, multiplied by 0.36, and this result by 100, gives the percentage of Magnesia (MgO).

Determination of the Alkalies, Soda (Na_2O), and Potash (K_2O):

7. The Silicate is decomposable, and the Silica may be expelled by Hydrofluoric and Sulphuric Acids.

Weigh 1 gram into a platinum dish, and cover it with a little water. Add drop by drop—stirring at the same time with a platinum wire—sufficient Hydrofluoric Acid to dissolve it, and then add a few drops of Sulphuric Acid. Evaporate to dryness on a sand bath, and heat over a Bunsen burner until no more white fumes are seen. Add about 5 Cc. strong Hydrochloric Acid, and boil until dissolved. Then add 10 Cc. of solution of Barium Chloride, and filter through a 11 Cm. filter-paper, allowing the filtrate to run into a 50 Cc. beaker. Wash thoroughly with hot

water. To the filtrate in the beaker add strong Ammonia and Ammonium Carbonate. Boil for a short time and filter into a platinum dish. Wash with hot water. Evaporate the filtrate in the dish to dryness, and heat over the flame until no more white fumes of Ammonium Chloride are given off. Dissolve in a little hot water, and add about 10 Cc. of Ammonium Carbonate and filter into a weighed platinum dish. Wash with hot water, and evaporate to dryness, heat over the direct flame to drive away all Ammonium Chloride, place in a dessicator and weigh when cool. The contents of the dish are Sodium Chloride and Potassium Chloride. In heating the dish over the direct flame, be careful to apply the heat gradually or the contents of the dish will spatter and thus cause loss.

8. Dissolve the Chlorides in the dish in the least possible quantity of hot water and add an equal volume of Platinic Chloride Solution. Place the dish on a water-bath and evaporate until the contents are nearly dry. Remove from the bath and allow them to dry spontaneously. Filter through a weighed Gooch crucible (Fig. 32) and wash with dilute Alcohol (1 in 2) until the washings running into the filtering flask are colorless. Place the Gooch crucible in a drying oven heated to 100° C. (not over) and when dry, remove to a dessicator and weigh. The contents of the Gooch crucible are Potassium Platinic Chloride (K_2PtCl_6). The weight of the K_2PtCl_6, multiplied by 148 and divided by 482, gives the weight of Potassium Chloride (KCl), this is to be subtracted from the weight of the mixed Chlorides obtained according to paragraph 7, and the result will be Sodium Chloride (NaCl). The weight of the NaCl just found, multiplied by 62 and divided by 116, gives the weight of Soda (Na_2O), and this multiplied by 100, gives the percentage. The weight of the K_2PtCl_6, multiplied by 94 and divided by 482, gives the weight of Potash (K_2O), and this multiplied by 100 gives the percentage.

9. The Silicate is undecomposable by treatment with Hydrofluoric and Sulphuric Acids.

Method of J. Lawrence Smith:

Weigh 1 gram of the finely-pulverized sample and place it on a sheet of black glazed paper. Grind up 1 gram of c. p. Ammonium Chloride and mix it on the paper with the sample. When well mixed, add about 8 grams of c. p. Calcium Carbonate and mix all

together very thoroughly. It is well to add the Calcium Carbonate a little at a time to insure a perfect mixture. Brush the whole into a platinum crucible, place the cover on and heat over a Bunsen burner. Heat very gently, just keeping the bottom red hot. When no more white fumes are given off raise the heat, so that the flame plays on the lower half of the crucible and continue thus for an hour. Allow the crucible to cool and remove the semi-fused mass with a glass rod, letting it fall into a 300 Cc. casserole. Wash off the cover and wash the crucible into the casserole, using a jet of hot water. Add enough hot water to make the volume about 50 Cc. and place the casserole on a water-bath. Cover it with a watch glass and heat for about a half hour. The mass will slake and crumble, and the alkalies will go into solution as chlorides. (In case the mass fuses together when in the platinum crucible, do not use force in detaching it, but place the crucible and cover in the casserole and add the hot water.) After heating on the water-bath, allow to stand over night. If in a hurry, proceed at once. Filter through a 11 Cm. filter-paper into a platinum dish and wash with hot water until no reaction is observed with Silver Nitrate Solution. To the filtrate in the dish add 25 Cc. of Ammonium Carbonate Solution and evaporate on the water-bath until the volume is about 20 Cc. Add 15 Cc. more of Ammonium Carbonate Solution and filter into a beaker. Wash three times with hot water, and to the filtrate in the beaker add 15 Cc. of Ammonium Carbonate Solution. Filter into a weighed platinum dish and when this dish is three-quarters full, substitute a beaker and place the dish on a water-bath. As the contents evaporate, add more filtrate, until the solution has all been filtered. Wash three times with hot water and pour the washings into the platinum dish. Evaporate to dryness and ignite, as described in paragraph 7. Weigh the dish and contents to get the weight of the mixed chlorides of Sodium and Potassium. Then finish exactly as directed in paragraph 8.

Clay Analysis.

Determination of Moisture:

Weigh 1 gram into a weighed platinum dish, place it in a drying oven heated to 100° C. and proceed exactly as described in Soda Ash analysis, p. 57.

Determination of Combined Water:

Weigh 1 gram into a weighed platinum crucible, place over a

blast lamp and ignite to constant weight exactly as described in Lime analysis, page 72, and deduct the percentage of moisture as found above.

Determination of Silica, Alumina, Ferric Oxide, Lime and Magnesia:

Proceed exactly as described in Typical Silicate Analysis, paragraphs 1, 2, 3, 4, 5 and 6. The Alumina precipitate, paragraph 2, will be large and difficult to properly wash. It should be poured, a little at a time, on the filter-paper, using a strong suction from the filter pump. Get as much as possible of the supernatant liquid from the beaker before pouring on the precipitate. When all is on the filter-paper, shut off the suction, fill the funnel with hot water, allow it to stand a minute and then remove the suction. Use a "policeman" to get the precipitate from the beaker.

In some clays, generally the light-colored ones, the Iron is present in the *Ferrous* state, and should be reported as Ferrous Oxide (FeO).

Determination of the Alkalies, Soda and Potash:

Proceed exactly as directed in paragraph 9.

Examples of Clay Analysis:

China Clay or Kaolin.	Per cent.	German Pot Clay.	Per cent
Silica, SiO_2,	45.55	Silica, SiO_2,	68.60
Alumina, Al_2O_3,	39.18	Alumina, Al_2O_3,	20.64
Soda, Na_2O,	.53	Ferrous Oxide, FeO,	.95
Potash, K_2O,	2.09	Lime, CaO,	.52
Combined Water, H_2O,	11.29	Magnesia, MgO,	.11
Moisture, H_2O,	.81	Soda, Na_2O,	.99
	——	Potash, K_2O.	.32
	99.45	Combined Water, H_2O,	7.75
			99.88

In judging the value of Clay from an analysis, the purpose for which it is intended must be borne in mind. When Clay is used as a constituent of the batch, as in the case of Opal glass made from Kaolin, the presence of Alkalies is not deleterious, but that of Iron in any form is objectionable. Where clay is used subjected to furnace heat, as in the form of fire brick, etc., the valuable constituents are Silica and Alumina. The others, Lime, Magnesia, Iron, and the Alkalies should be of as low percentage as possible because they are fluxes.

Fire Brick.

Determination of Silica, Alumina, Ferric Oxide, Lime and Magnesia:

Proceed exactly as directed in Typical Silicate Analysis, paragraphs 1 to 6 inclusive.

Determination of the Alkalies:

For a Silica brick proceed according to paragraphs 7 and 8, for an Alumina brick according to paragraph 9.

Examples of Fire Brick Analysis:

Silica Brick—Abernnant Brand.	Per cent.	Alumina Brick—Queen's Run Brand.	Per cent.
Silica, SiO_2,	91.90	Silica, SiO_2,	52.17
Alumina, Al_2O_3,	3.31	Alumina, Al_2O_3,	45.58
Ferric Oxide, Fe_2O_3	.53	Ferric Oxide, Fe_2O_3,	none
Lime, CaO,	2.35	Lime, CaO,	none
Magnesia, MgO,	.29	Magnesia, MgO	none
Soda, Na_2O,	.77	Soda, Na_2O,	.84
Potash, K_2O,	.36	Potash, K_2O,	.97
	99.51		99.56

Feldspar.

Determination of Silica, Alumina, Ferric Oxide, Lime and Magnesia:

Proceed exactly as directed in Typical Silicate Analysis, paragraphs 1 to 6, inclusive.

Determination of the Alkalies:

Proceed as directed in paragraph 9.

Determination of Combined Water:

Weigh 1 gram of the finely-pulverized mineral into a weighed platinum crucible and ignite over a blast lamp to constant weight.

Example of a Feldspar Analysis:

Silica, SiO_2,	59.20 per cent.
Alumina, Al_2O_3,	18.00 "
Ferric Oxide, Fe_2O_3,	none.
Soda, Na_2O,	4.77 "
Potash, K_2O,	9.48 "
Combined Water, H_2O,	8.75 "
	100.20 "

Sand.

The impurities to be determined are Alumina (Al_2O_3), Iron as Ferric Oxide (Fe_2O_3), Moisture (H_2O) and Organic Matter.

Sand may be analyzed according to Typical Silicate Analysis, paragraphs 1 to 4 inclusive, but owing to the large proportion of Silica (SiO_2) and the small proportion of the impurities, this method is liable to error, unless performed by a skilled operator. It is customary to determine the impurities and subtract their sum from 100 and call the difference Silica.

Determination of Alumina and Ferric Oxide:

Finely pulverized so that no gritty particles remain when tested by rubbing with the finger. Weigh 2 grams into a platinum dish and add enough cold water to cover the sand. Add, drop by drop, sufficient Hydrofluoric Acid to dissolve, stirring at the same time with a platinum wire. Add a few drops of Sulphuric Acid and evaporate to dryness on a sand-bath. Heat over the naked flame until all white fumes have ceased coming off. Add about 5 Cc. concentrated Hydrochloric Acid and one drop of concentrated Nitric Acid and boil until the contents of the dish are dissolved. Pour into a 50 Cc. graduated flask and wash the dish into the flask with cold water. Cool the flask by running hydrant water over the outside and fill to the mark with cold water. Insert the stopper and mix by shaking.

With a 25 Cc. pipette take out that volume and run it into a 50 Cc. beaker. Empty the flask into a similar beaker and wash it and the pipette into this last beaker, using hot water. Precipitate the Alumina and Ferric Oxide with strong Ammonia, as described in paragraph 2, and determine them according to paragraphs 3 and 4. In determining the Ferric Oxide, use Weak Standard Permanganate.

Determination of Moisture (H_2O):

Weigh 5 grams of the sand (not pulverized) into a weighed platinum dish and heat in a drying oven at 100° C. to constant weight. The loss of weight will be moisture, which, multiplied by 20, will give the percentage.

Determination of Organic Matter:

Weigh 5 grams (not pulverized) into a weighed platinum crucible. Ignite for 15 minutes over the blast lamp. Cool in a dessicator and weigh. The loss of weight will be organic matter plus

moisture. Subtract the moisture as found above. The difference, multiplied by 20, will give the percentage of organic matter.

Simple Tests.

Examine the sand with a lens. The grains should be sharp. Bubbles in the grains are not objectionable, as they contain gas—probably CO_2—which expands when the sand is heated, thus splitting up the grains and promoting fusion.

The sand should be colorless and remain so after heating. Ignite a little in a platinum crucible and examine again with the lens. It should not become colored, as this indicates the presence of oxidizable impurities, as Iron, etc.

Iron may be present as Ferrous Silicate, Ferric Silicate, Magnetic Oxide (Fe_3O_4), and in some sands, as the mineral Mennacanite. The Silicate of Iron cannot be dissolved out. The Magnetic Oxide may be removed by treatment in a magnetic separator.

Fig. 80 A.

When sand is treated with Hydrochloric Acid it should not effervesce, as this would indicate the presence of carbonates, as Lime, etc.

Sand should be entirely free from Cobalt compounds. These may be tested for by placing the finely-ground sand in a platinum crucible, adding water, Hydrofluoric and Sulphuric Acids, evaporating to dryness, igniting, to drive off all the white fumes, and then adding a little Borax and fusing to a glassy mass. If Cobalt is present, the mass will be of a deep blue color.

To determine if a sand has been properly washed, place a little in a test tube (Fig. 80 A) and half fill the tube with cold water. Place the thumb on the open end and shake vigorously. Allow the sand to settle. The clay present will remain suspended in the water. The greater the amount of clay present the more turbid will the water appear.

Example of a Sand Analysis.

Juniata Sand, 2 grams taken, treated with HF and H_2SO_4, ignited, and residue dissolved in HCl. Solution divided into two parts, each equivalent to 1 gram.

Determination of Fe_2O_3:

Weak Standard Permanganate, 0.3 Cc.

$0.3 \times .0014 = .00042$

$.00042 \times 100 = .042$ per cent. Fe_2O_3.

Determination of Al_2O_3.

Weight of crucible, cover, $Al_2O_3 + Fe_2O_3$,	43.71660
Weight of crucible and cover,	43.71580
Weight of $Al_2O_3 + Fe_2O_3$,	.00080
Weight of Fe_2O_3 found above,	.00042
Weight of Al_2O_3,	.00038

$.00038 \times 100 = .038$ per cent. Al_2O_3.

Determination of Moisture (H_2O), 5 grams taken:

Weight of dish and sand before drying,	23.5892
Weight after drying,	23.5871
Loss of weight, or Moisture,	.0021

$.0021 \times 20 = .042$ per cent. H_2O.

Determination of Organic Matter, 5 grams taken:

Weight of crucible and sand before igniting,	48.7162
Weight after igniting,	48.7070
Loss of weight, or Moisture plus Organic Matter,	.0092
Moisture as found above,	.0021
Difference, or Organic Matter,	.0071

$.0071 \times 20 = .142$ per cent. Organic Matter.

Summary.

Silica, SiO_2—by difference,	99.736	per cent.
Alumina, Al_2O_3,	.038	"
Ferric Oxide, Fe_2O_3,	.042	"
Moisture, H_2O,	.042	"
Organic Matter,	.142	"
	100.000	"

Glass Analysis.

Break the glass up in a steel mortar (Fig. 6) and empty the

mortar into a porcelain dish. Pass a magnet through the broken glass to remove any particles of steel abraded from the mortar. Finish the pulverization in an Agate mortar (Fig. 4) getting the powder to the consistency of flour, having no gritty particles that may be felt by the finger. Place the powder in a porcelain dish, and dry in an oven for about a half-hour. When dry place it in a tightly stoppered vial (Fig. 10). Use this dried sample for the analysis, taking care to keep the vial tightly stoppered when not in use, as powdered glass absorbs moisture from the air.

Determination of Silica, Alumina and Ferric Oxide, Lime and Magnesia:

Proceed exactly as directed in Typical Silicate Analysis, paragraphs 1 to 6 inclusive. Unless it is desired to find the percentages of Alumina and Ferric Oxide separately, omit the division into two parts of the filtrate from the Silica, paragraph 2, and omit also the determination of Ferric Oxide, paragraph 3, proceeding directly to paragraph 4, determining the Al_2O_3 and Fe_2O_3 together, and multiplying their combined weights by 100 to get the percentage. Determine Lime and Magnesia as directed in paragraphs 5 and 6.

Determination of the Alkalies, Soda and Potash:

Proceed according to paragraphs 7 and 8.

Examples of Glass Analysis.

Common Bottle-Glass:	
Silica, SiO_2,	71.50
Alumina and Ferric Oxide, $Al_2O_3 + Fe_2O_3$,	1.24
Lime, CaO,	11.35
Soda, Na_2O,	15.98
	100.07 per cent.
Lime Flint-Glass:	
Silica, SiO_2,	73.68
Alumina and Ferric Oxide, $Al_2O_3 + Fe_2O_3$,	2.07
Lime, CaO,	3.85
Magnesia, MgO,	2.78
Soda, Na_2O,	17.34
	99.72 per cent.

Bohemian Glass:

Silica, SiO_2,	72.22
Alumina and Ferric Oxide, $Al_2O_3 + Fe_2O_3$,	1.46
Lime, CaO,	7.98
Soda, Na_2O,	9.53
Potash, K_2O,	8.32
	99.51 per cent.

Lead Glass.

Determination of Silica (SiO_2), Lead Oxide (PbO), Alumina and Ferric Oxide ($Al_2O_3 + Fe_2O_3$).

Proceed as directed in Typical Silicate Analysis, paragraph 1, using Nitric Acid instead of Hydrochloric Acid to dissolve the fused mass. Evaporate the Nitric Acid solution to dryness, break up the residue in the casserole with the glass rod (Fig. 80), and redissolve in the smallest sufficient quantity of strong Nitric Acid. Evaporate again to dryness to render the Silica (SiO_2) insoluble. Add about 50 c.c. of dilute Nitric Acid, heat on the air or water bath with constant stirring. Filter through a 11 c.m. ashless filter paper. Wash the filter thoroughly with hot water. Remove it to a weighed platinum crucible. Ignite over a blast lamp until the contents are white. Remove the crucible to a dessicator and weigh when cool. Then add a little water to the contents of the crucible, some Hydrofluoric Acid, and a few drops of c. p. strong Sulphuric Acid. Evaporate carefully over a Bunsen burner, and when white fumes have ceased to come off, raise the heat and continue heating until the contents of the crucible are white. Cool and weigh again. The loss of weight will be Silica (SiO_2). Set the crucible aside for the present.

To the filtrate from the SiO_2 precipitation add c. p. strong Sulphuric acid until no more precipitate forms. Boil gently and filter through a weighed Gooch crucible (Fig. 33) prepared as described on page 20. Wash with hot water until the washings are neutral to litmus paper. Ignite over a Bunsen burner, cool, and weigh. This gives the weight of Lead Sulphate ($PbSO_4$) which multiplied by 221 and divided by 301 gives the weight of Lead Oxide (PbO).

To the contents of the platinum crucible which contains the residue from the Silica determination, add a little Silica flux (page

46) and heat to fusion. Dissolve in a little dilute Hydrochloric Acid and add this solution to the filtrate from the Lead Sulphate precipitation, and treat this combined solution according to paragraphs 2, 3, and 4.

Determination of Soda and Potash.

Treat the glass according to paragraph 7, and ignite as usual until all the white fumes are driven off. Add about 10 c c. of dilute Hydrochloric Acid, boil for a short time and filter through a weighed Gooch crucible as described above. Wash with hot water, add Barium Chloride solution to the filtrate, and from this point proceed according to paragraph 7, to determine the Alkalies.

Note.—The Gooch crucible and contents may be weighed and the previous determination of Lead Oxide verified.

Example, 1 gram taken:

Determination of Silica (SiO_2).

Insoluble matter from paragraph 1, ignited and weighed in a platinum crucible, treated with HF and H_2SO_4, ignited and weighed again.

Weight before adding HF and H_2SO_4,	56.1526
Weight after adding HF and H_2SO_4,	55.4448
Loss of Weight or Silica (SiO_2),	.7078

$.7078 \times 100 = 70.78$ per cent. SiO_2.

Filtrate from above precipitated with H_2SO_4, filtered, ignited, and weighed in Gooch crucible.

Weight of crucible and contents,	49.0213
Weight of crucible,	48.8329
Weight of Lead Sulphate ($PbSO_4$),	.1884

$.1884 \times \frac{2}{3}\frac{2}{0}\frac{1}{1} = .1383$; $.1383 \times 100 = 13.83$ per cent. PbO.

Filtrate from paragraph 1.

Weight of whole,	55.4125
Weight of crucible,	55.4082
Weight of $Al_2O_3 + Fe_2O_3$,	.0043

$.0043 \times 100 = 0.43$ per cent. $Al_2O_3 + Fe_2O_3$.

Paragraph 7.

Determination of the Alkalies, Potash and Soda, 1 gram taken:

Weight of platinum dish and chlorides,	50.3457
Weight of platinum dish,	50.0925
Weight of chlorides (KCl and NaCl),	.2532
Weight of Gooch crucible and K_2PtCl_6,	42.8356
Weight of Gooch crucible,	42.5836
Weight of K_2PtCl_6,	.2520

$0.2520 \times \frac{148}{482} = .0774$ KCl.

$0.2520 \times \frac{94}{482} = .0491$ K_2O; $.0491 \times 100 = 4.91$ per cent. Potash.

Weight of chlorides as found above,	.2532
Weight of KCl as found above,	.0774
Weight of NaCl,	.1758

$.1758 \times \frac{62}{116} = .0939$; $.0939 \times 100 = 9.39$ per cent. Soda.

Summary.

Silica, SiO_2,	70.78
Lead Oxide, PbO,	13.83
Alumina and Ferric Oxide, $Al_2O_3 + Fe_2O_3$,	.43
Potash, K_2O,	4.91
Soda, Na_2O,	9.39
	99.34 per cent.

Baryta Glass.

Determination of Silica (SiO_2):

Proceed as directed in Typical Silicate Analysis, paragraph 1.

Determination of Barium Oxide (BaO):

Boil the filtrate from the Silica and add about 1 Cc. of hot dilute Sulphuric Acid. Filter, ignite and weigh the precipitated Barium Sulphate. Multiply its weight by 152 and divide by 232 to get the weight of Barium Oxide. This multiplied by 100 gives the percentage.

Determination of Al_2O_3 and Fe_2O_3:

Add strong Ammonia in excess to the filtrate from the Barium Sulphate precipitation; boil, filter off the Aluminium and Ferric

Hydroxides, ignite in a weighed porcelain crucible and weigh. The weight of $Al_2O_3 + Fe_2O_3$, multiplied by 100, gives the percentage.

Determination of Alkalies:

Proceed as directed in paragraph 7. Boil the insoluble residue left after treatment with HF and H_2SO_4 in dilute Hydrochloric Acid. Filter, and if desired, ignite and weigh the filter—which is Barium Sulphate—to check the former Barium Oxide determination. The filtrate is then treated according to the remainder of paragraphs 7 and 8.

Zinc Glass.

Determination of Silica (SiO_2):

Proceed according to paragraph 1, Typical Silicate Analysis.

Determination of Al_2O_3 and Fe_2O_3:

Add to the filtrate from the Silica just enough c. p. dry Sodium Carbonate to make it neutral to Litmus paper. Heat to boiling and while boiling add 10 Cc. of Sodium Acetate Solution. Boil for a few minutes, filter, wash, dry, ignite in a weighed porcelain crucible and weigh as $Al_2O_3 + Fe_2O_3$.

Determination of Zinc Oxide (ZnO):

To the filtrate obtained from the $Al_2O_3 + Fe_2O_3$ precipitation, add sufficient Acetic Acid to give a slight odor. Boil, and while boiling, pass Hydrogen Disulphide gas through the solution for about a half hour. Filter off the precipitated Zinc Sulphide, wash it with hot water. Ignite it in a weighed porcelain crucible and weigh as Zinc Oxide (ZnO).

Determination of Lime and Magnesia:

Boil the filtrate from the Zinc precipitation until the odor of H_2S has gone and proceed as directed in paragraphs 5 and 6.

Determination of the Alkalies:

Proceed according to paragraph 7, but before adding Ammonium Carbonate, add Ammonia and Ammonium Sulphide, to precipitate the Zinc. Then filter this off, wash with hot water, boil the filtrate, add Ammonia and Ammonium Carbonate and continue as directed in paragraphs 7 and 8.

Example of a Zinc Glass Analysis.

Silica, SiO_2,	71.12
Alumina and Ferric Oxide, $Al_2O_3 + Fe_2O_3$,	1.03
Zinc Oxide. ZnO,	2.38
Lime, CaO,	5.65
Magnesia, MgO,	3.43
Soda, Na_2O,	16.83
	100.44 per cent.

Determination of Manganese Oxide in Glass:

Precipitate the Al_2O_3 and Fe_2O_3 with Sodium Acetate, as directed in Zinc Glass, filter them off and wash with hot water. Boil the filtrate, and while boiling, add a few drops of Bromine. Continue boiling and stirring for a short time. The Manganese will separate as MnO_2. Filter it off. Wash with hot water, ignite in a weighed porcelain crucible and weigh as Mn_3O_4, which, multiplied by 211 and divided by 227, gives the weight of MnO.

Calculation of Recipe from the Analysis of Glass.

Suppose, for example, we take the analysis of common bottle glass, given on page 92. The ingredients to be considered are Silica 71.5 per cent., Lime 11.35 per cent. and Soda 15.98 per cent. Multiply each percentage by 100 and divide the result by the percentage of Silica:

Silica, $71.5 \times 100 \div 71.5 = 100$ parts.
Lime, $11.35 \times 100 \div 71.5 = 15.9$ parts.
Soda, $15.98 \times 100 \div 71.5 = 22.3$ parts.

Convert the constituents into the equivalent raw materials. This is done by means of knowledge gained from previous analysis of the raw materials to be used.

Silica may be considered as Sand, 100 parts. Lime (CaO) must be converted into either Carbonate ($CaCO_3$) or Hydroxide (Slaked Lime), $Ca(OH)_2$.

To convert the CaO into $CaCO_3$, multiply by 100 and divide by 56.

Example:

CaO, $15.9 \times 100 = 1590 \div 56 = 28.4$ parts.

To convert the CaO into $Ca(OH)_2$, multiply by 74 and divide by 56.

Example:

$$CaO,\ 15.9 \times 74 = 1176.6 \div 56 = 21 \text{ parts.}$$

If Soda Nitrate has been used in the glass, part of the Na_2O will be due to it, and as there is no method for determining the percentage of Na_2O due to the Nitrate originally used, the glassmaker must deduct from the proportion of Na_2O the amount of Na_2O in the usual amount of Nitrate that he is accustomed to employ. To find the percentage of Soda (Na_2O) in a given amount of Nitrate ($NaNO_3$), multiply this amount by 62 and divide by 170.

Example:

Suppose 5 parts of Nitrate were to be used, $5 \times 62 = 310 \div 170 = 1.8$ parts Na_2O, to be deducted from the Na_2O found by the analysis, we have $22.3 - 1.8 = 20.5$ parts Na_2O due to Soda Ash. Calculate this to Sodium Carbonate, multiply by 106 and divide by 62:

$$20.5 \times 106 = 2373 \div 62 = 36.8 \text{ parts } Na_2CO_3.$$

As c. p. Sodium Carbonate contains 58.49 per cent. Soda (Na_2O), we may consider the 36.8 as 37 parts of 58 per cent. Soda Ash. If the Soda Ash to be added is 48 per cent., we may increase the 36.8 per cent. by 10 per cent., as $36.8 + 3.7 = 40.5$ parts of 48 per cent. ash.

The calculation may be summed up as follows:

Sand,	100	Sand,	100
Soda Ash, 48 per cent.,	40	Soda Ash, 48 per cent.,	40
Lime Carbonate,	28	Slaked Lime,	21
Nitrate,	5	Nitrate,	5

The calculation will only give an approximate recipe, the final correct recipe must be obtained by the glassmaker's knowledge of his art.

Batch Analysis.

A rapid approximate method for determining the proportions of a simple batch (A. H. Elliott):

Weigh 10 grams of the batch into a *weighed* 250 Cc. graduated flask. Add about 100 Cc. of cold water and shake well. Allow the flask to stand about half an hour, shaking at intervals. Fill to the mark with cold water, insert the stopper and mix well by shaking. Allow to settle. Take out 25 Cc. of the supernatant liquid with a graduated pipette and allow it to run through a filter-

paper into a 100 Cc. Erlenmeyer flask. Wash the filter twice with hot water. Remove the Erlenmeyer flask to a burette, add a drop of Methyl Orange Indicator and titrate with Standard Acid as in Soda Ash Analysis, page 53. The 25 Cc. taken, being one-tenth of the original volume, contains the Soda Ash in 1 gram of the batch. The number of Cc. and tenths of the Standard Acid used, multiplied by .053, and this result by 100, gives the percentage of 58 Soda Ash present.

Pour the contents of the 250 Cc. graduated flask through the same filter into another flask and set this filtrate aside, to be used in case another or check determination of Soda Ash is to be made. Place a beaker under the funnel and pour a little hot water into the 250 Cc. graduated flask. Shake it with a rotary motion and then allow the sand to settle. Pour this water into the funnel, keeping the sand in the flask. Repeat this washing twice. Then wash the filter-paper by filling the funnel twice with hot water. Remove the beaker from beneath the funnel and substitute for it the 250 Cc. graduated flask. With a pipette, allow 60 Cc. of Standard Acid to run on the filter-paper and when this has passed into the flask, make a hole in the bottom of the paper with a glass rod and with hot water wash the contents of the paper down into the flask. Shake the flask until effervescence has ceased, and when the Lime has dissolved, fill the flask to the mark with cold water. Insert the stopper and mix by shaking. Allow the sand to settle and take out with a pipette 100 Cc. of the supernatant liquid and run it into a 250 Cc. Erlenmeyer flask. This 100 Cc. contains the Lime in 4 grams of the batch. Add a drop of Methyl Orange Indicator to the flask, place the flask under a burette and run in enough Standard Sodium Carbonate Solution to cause the pink color just to appear. The number of Cc. and tenths of Standard Sodium Carbonate subtracted from the volume of Standard Acid ($60 \times \frac{4}{10} = 24$ Cc. used in the 100 Cc. taken) leaves the volume of Acid neutralized by the lime in 4 grams of batch. This volume of Acid, multiplied by .028, gives the amount of Lime in the 4 grams, and this last result, multiplied by 25, gives the percentage of Lime.

Pour the liquid from the flask slowly and carefully without allowing any of the sand to escape. Half fill the flask with hot water, gently shake with a rotary motion. Allow to settle, and pour out the liquid as before. Repeat this washing of the sand.

Place the flask in a drying oven, heated to 100° C., and allow it to remain until the sand is dry, then remove it and insert a glass tube; apply the mouth to the upper end of this tube, and suck out the steam. Allow the flask to cool, and weigh it. Deduct the weight of the flask. The difference will be the weight of the sand in the 10 grams of the batch taken. Multiply this by 10 to get the percentage.

Example of a Batch Analysis, 10 grams taken:

25 Cc. of Solution containing Soda Ash in 1 gram of Batch. Standard Acid used 4.6 Cc.

$4.6 \times .053 = .2438 \times 100 = 24.38$ per cent. Soda Ash.

60 Cc. of Standard Acid used to dissolve the Lime in 10 grams of Batch. 100 Cc. of the total solution, equal to 4 grams of Batch, containing $\frac{4}{10}$ of 60 Cc. or 24 Cc.

Standard Acid,	24. Cc.
Standard Sodium Carbonate,	14.3 Cc.
Difference, or Standard Acid used for Lime in 4 grams of Batch,	9.7 Cc.

$9.7 \times .028 = .2716 \times 25 = 6.79$ per cent. Lime (CaO).

Weight of flask and sand,	45.188
Weight of flask	39.028
Weight of Sand,	6.160

$6.16 \times 10 = 61.60$ per cent. Sand.

Calculation of Batch.

Proceed as directed on page 97.

Sand, $61.6 \times 100 \div 61.6 = 100$ parts.
Soda, $24.38 \times 100 \div 61.6 = 39$ parts.
Lime, $6.79 \times 100 \div 61.6 = 11$ parts.

The Lime may be calculated into Carbonate or Hydroxide as directed on page 97.

FUEL ANALYSIS.

Coal, Proximate Analysis.

Extract from the report of the Committee on Coal Analysis of the American Chemical Society, published December, 1899:

Sampling.—In sampling from cars proceed as follows: Beginning at one corner of the car drive a scoop-shovel vertically down as

deep as it will reach. Bring it out with all the coal it will hold, and throw into a cart or wheelbarrow. Repeat, taking six scoopfuls along one side of the car, at equal intervals, six through the center, and six along the other side. Place the coal taken on a close, tight floor. Break all lumps larger than an orange. Mix by shoveling it over on itself, back and forth. Quarter, and reject opposite quarters Break finer as may be necessary, and continue to quarter down until a sample is obtained small enough to go into a quart fruit jar, and having no pieces larger than one-fourth inch cube. The sample may, with advantage, be run through a mill which will break it to the size mentioned. Transfer to the jar and make sure the latter is sealed air-tight before it is set aside. All of these operations should be conducted as rapidly as possible to guard against any change in the moisture content of the coal.

Modifications of this method will of course suggest themselves, and in some cases will be necessary. When possible, a more representative sample may be secured by taking shovelfuls of the coal at regular intervals during the loading or unloading of the car. In any method of sampling, two conditions must be insisted on; the original sample should be of considerable size and thoroughly representative, and the quartering down to an amount which can be put in a sealed jar should be carried out as quickly as possible after the sample is taken.

In boiler tests shovelfuls of coal should be taken at regular intervals and put in a tight covered barrel, or some air-tight covered receptacle, and the latter should be placed where it is protected from the heat of the furnace.

For analysis quarter down further to about 100 grams. Run this portion through a mill which admits of quick grinding with little exposure to the air. A coffee mill set to grind as finely as possible will answer. The grinding of 100 grams is recommended because less moisture will be lost than if a smaller sample is ground. When an accurate determination of moisture is required and especially with coals high in moisture, a portion of this coarsely ground sample must be transferred at once to a tighty stoppered tube for use in determining moisture. Grind 12 or 15 grams of the remainder moderately fine in a porcelain or iron mortar and transfer to a tightly corked tube for use in other determinations than moisture.

Moisture.—Dry 1 gram of the coal in an open porcelain or platinum crucible at 104°–107° C. for one hour in a drying oven. Cool in a dessicator and weigh covered.

Volatile Combustible Matter.—Place 1 gram of the fresh, undried, powdered coal in a platinum crucible weighing 20 to 30 grams and having a tightly-fitting cover. Heat over the full flame of a Bunsen burner for 7 minutes. The crucible should be supported on a platinum triangle with the bottom 6 to 8 Cm. (about 3 inches) above the top of the burner. The flame should be fully 20 Cm. (8 inches) high, burning free, and the determination should be made in a place free from draughts. The upper surface of the cover should burn clear, but the under surface should remain covered with carbon. To find the Volatile Combustible Matter, subtract the percentage of moisture from the loss found here.

Ash.—Burn the powdered coal used for the determination of Moisture, at first over a very low flame, with the crucible open and inclined, until free from carbon. It is advisable to examine the ash for unburned carbon by moistening it with alcohol.

Fixed Carbon.—Subtract the percentage of Ash from the percentage of coke.

Example of a Coal Analysis.

Moisture, 1 gram taken:

Weight of crucible, cover and coal before drying,	59.6965
Weight after drying,	59.6800
Loss or Moisture,	.0165

$.0165 \times 100 = 1.65$ per cent. Moisture.

Volatile Combustible Matter, 1 gram taken:

Weight of crucible, cover and coal before igniting,	30.5793
Weight after igniting,	30.2187
Loss,	.3606
Moisture as found above,	.0165
Volatile Combustible Matter,	.3441

$.3441 \times 100 = 34.41$ per cent. Vol. Comb. Matter.

Ash, 1 gram taken:

Weight after driving off Moisture,	59.6800
Weight after burning away Carbon,	59.6057
Ash,	.0743

$.0743 \times 100 = 7.43$ per cent. Ash.

Fixed Carbon:

Weight after igniting,	30.2188
Weight of crucible and cover,	25.5793
Weight of coke,	.6394
Weight of ash,	.0743
Weight of fixed Carbon,	.5651

$.5651 \times 100 = 56.51$ per cent. Fixed Carbon.

Summary of Coal Analysis.

Moisture,	1.65	per cent.
Volatile Combustible Matter,	34.41	"
Fixed Carbon,	56.51	"
Ash,	7.43	"
	100.00	"

Coke.

Determination of Moisture, Carbon and Ash:

Moisture.—Weigh 1 gram into a weighed platinum crucible, dry in an air-bath at 104° to 107° C. The loss of weight will be moisture.

Carbon.—Place the crucible containing the dried coke over a Bunsen burner in an inclined position and heat until only ash is left. The loss of weight will be Carbon.

Ash.—Deduct the weight of the crucible, the result will be ash.

Example of Coke Analysis, 1 gram taken:

Weight of crucible and cover,	24.2725
Weight of crucible, cover and coke before drying,	25.2725
Weight after drying,	25.2631
Loss, or Moisture,	.0094

$.0094 \times 100 = 0.94$ per cent.

Weight of whole after drying,	25.2631
Weight after burning off the Carbon,	24.3586
Loss, or Carbon,	.9045
$.9045 \times 100 = 90.45$ per cent.	
Weight of whole after burning off Carbon,	24.3586
Weight of crucible and cover,	24.2725
Weight of Ash,	.0861
$.0861 \times 100 = 8.61$ per cent.	

Summary.

Moisture,	.94
Carbon,	90.45
Ash,	8.61
	100.00 per cent.

GAS ANALYSIS.

The Analysis of Gas is a very simple operation and furnishes an efficient means of control for the workings of the gas-producer. By means of analysis we are enabled to ascertain if a producer is being properly managed and the coal is being converted into the most valuable gases.

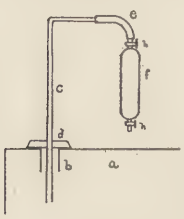


Fig. 81.

Sampling.—The sample of gas may be taken from the top of the producer. Figure 81 shows the method of sampling. The top of the producer *a* has a hole *b* into which is inserted the iron pipe *c*. The heavy iron plate *d*, fastened to the pipe, keeps it upright. A rubber tube *e* is permanently attached to the upper arm of the pipe. The pipe is placed in the hole, and the gas allowed to escape from the rubber tube. After a couple of minutes the gas tube *f*, filled with water, is attached to *e*. The stop-cocks *h h* are opened, and as the water escapes, the gas takes its place in the tube. When the water has all run out, the stop-cocks are closed and the tube removed. The iron pipe is then withdrawn, and the hole *b* closed with a plug.

Fig. 82 shows the gas tube separately. The bore of the ends *k k* and stop-cocks *h h* should be as fine as possible, so as to prevent

any air lodging in them and passing into the tube along with the gas. Capillary glass tubing should be used for these parts of the

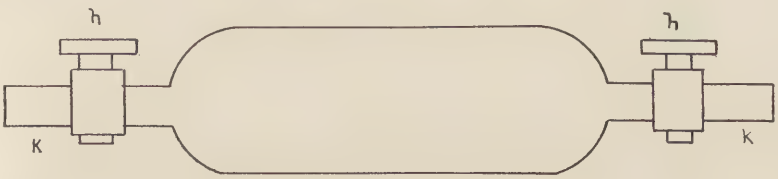


Fig. 82.

sample tube. The total length of the sample tube should be abou 12 inches, and the diameter of the cylindrical part about 2 inche: outside and about $\frac{1}{8}$ inch thick.

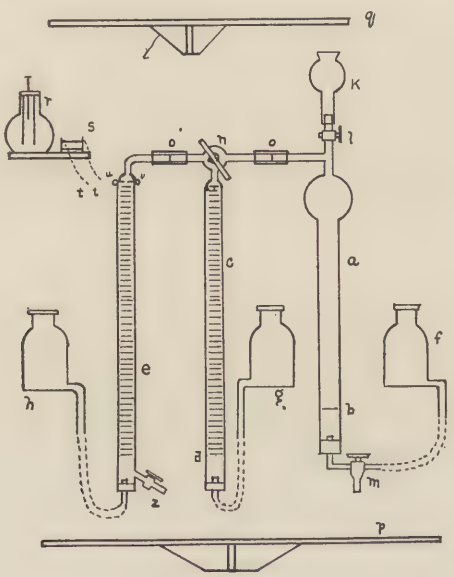


Fig. 83.

Apparatus.—Figure 83 represents an excellent apparatus, designed by Dr. Arthur H. Elliott. The tube *a* is of about 125 Cc. capacity. It has a mark at *b*, indicating 100 Cc. The tube *c* is graduated in Cc. and tenths, and has a capacity of 100 Cc. to the

mark *d*. The explosion tube *e* is also graduated. These tubes are connected with aspirator bottles *f*, *g* and *h* by means of rubber tubing. The tube *a* has a detachable funnel *k* for the introduction of reagents, and it is fitted with a one-way cock *l* and a two-way cock *m*. The tube *c* is fitted with a three-way cock *n*, which permits connecting *a* with either *c* or *e*. The ends of the tubes are brought together and joined by pieces of heavy rubber tubing *o*, *o*. The whole apparatus may be supported by means of clamps in a similar manner to which a burette is suspended.

Reagents.—For the analysis of producer gas, the following are required:

Potassium Hydroxide, to absorb Carbon Dioxide: About 50 grams, dissolved in 250 Cc. of distilled water and kept in a bottle with a rubber stopper.

Potassium Pyrogallate, to absorb Oxygen. To a solution of Potassium Hydroxide of the above strength, add about 8 grams of Pyrogallic Acid and keep in a similar bottle.

Cuprous Chloride, to absorb Carbonic Oxide (CO). A saturated solution in concentrated Hydrochloric Acid and containing a few pieces of clean copper wire. Keep in a glass-stoppered bottle.

Oxygen Gas, for the explosion in determining Hydrogen and Marsh Gas, if the percentages of these constituents are required. Oxygen may be purchased in Iron cylinders ready for use.

Operation.—First fill the apparatus with water. To do this, place the bottles *f*, *g* and *h* (Fig. 83) on the shelf *p*, and fill them about two-thirds full of water. Then open the three-way cock *n* so as to connect *e* with *a*. Place the bottle *h* on the shelf *q*. The water will rise in *e*. When it has reached the junction of *a*, turn *n* to connect *c* and *a*, and place the bottle *g* on the shelf *q*. When *c* is full of water, shut off *n* entirely. Open *m* so that *f* is connected with *a* and place the bottle *f* on the shelf *q*. Then open the cock *l* until the water reaches the funnel *k*. The apparatus is now ready for the introduction of 100 Cc. of the gas to be tested.

Figure 84 shows the manner in which this is performed. The gas tube *v* is connected with the aspirator *w*, by means of a piece of flexible tubing *x*. The flexible tube *y* is attached to the other end of *v* and held in a vertical position, as shown by the dotted lines. The funnel *k* (Fig. 83) is detached and laid aside. The bottle *f* is placed on the lower shelf *p* and the cock *m* opened. The

cocks of v are then opened and a little water permitted to enter v to expel the gas and drive any air out of y. The upper cock of v is closed and the upper end of y pinched with the finger, y is then bent over and connected with the upper end of a. The upper cock of v is again opened. The cock l is then opened and the gas allowed to enter a. When the level of the water in a is a little below the mark b, the cock l is closed. The tube y is detached from a and the bottle f raised until the surface of its contents are on the same level as the water in a. If these levels are at the mark b, the

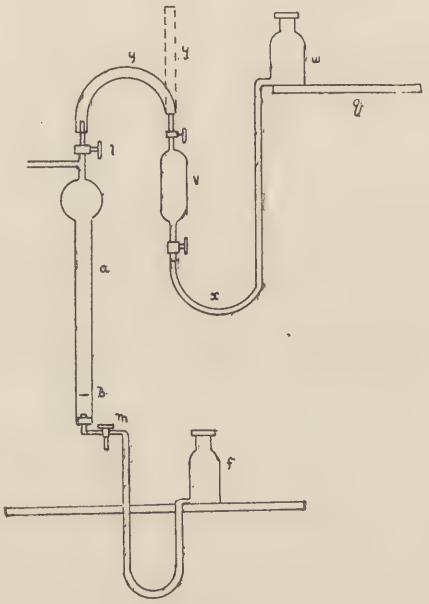


Fig. 84.

proper volume of gas is contained in a. If above b, more gas must be admitted from y. If below b, some gas must be expelled by cautiously opening l. The water in a will rise; l is closed when the mark is reached. The funnel k is then replaced on a.

Analysis.—Place the bottles f and g (Fig. 83) on the shelf p. Half fill the funnel k with the Potassium Hydrate Solution. Cautiously open l and permit the solution to gradually enter a, allowing it to run down the inner surface. When k is *nearly* empty, close the stop-cock l and wait a few minutes. Place the

bottle *f* on the shelf *q*, open the stop-cock *n* so that *a* is connected with *c*, and permit the water to rise in *a* until it reaches *n*. Close *n* and raise *g* until the surface of its contents are level with the water in *c*. Observe the number of Cc. and tenths shown on *c*. This reading will be the percentage of Carbon Dioxide (CO_2). Turn the cock *m* so that it delivers through the bottom, open *l* and permit *a* to empty through *m*. When empty, fill *k* with water and allow it to run through *a*, washing out the tube. Connect *a* and *f* by means of *m*, and allow the water to rise in *a* until it enters the funnel, then close *l*. Replace *f* on the shelf *p*, and *g* on the shelf *q*. Open *n* and allow the water to rise in *c*, driving the gas into *a*. Then close *n*. Fill *k* with the Pyrogallate Solution, and allow it to run into *a*. Place *f* on the shelf *q*, and *g* on *p*. Open *n*, and drive the gas into *c*. Take the reading as before. The loss of volume will be Oxygen (O). After again washing out *a* and filling it with water, pass the gas from *c*, and add Cuprous Chloride Solution by means of *k*, and then fill *k* with water and add this also. Pass the gas into *c* and take the reading. The loss will be Carbonic Oxide (CO).

After proceeding thus far, the residue in *c* may consist of Hydrogen, Marsh Gas, and Nitrogen. If it is desired to determine these we make use of the explosion tube *e*. Place the bottle *h* on the shelf *p* and *g* on *q*. Turn the cock *n* so that *c* connects with *e*, and pass the gas into *e*. Allow about 20 Cc. of the gas to enter *e* and close *n*. Raise the bottle *h* until the surface of its contents is level with the water in *e* and take the reading. Connect the stop-cock *z* by means of a rubber tube with the cylinder of Oxygen Gas, and allow about 75 Cc. of this gas to enter *e*. Take the reading of *e* again, and place *h* as low as possible. Wait a few minutes for the gases to mix. Connect the wires *t*, *t*, from the Rhumkorff coil *s* with the platinum loops *u u*, and by means of the battery *r* pass a spark. The mixture in *e* will explode, and its volume be diminished. Wait a few minutes and then raise the bottle *h* as before and take the reading. While waiting fill *a* with water. Place *h* on the shelf *q*, and *f* on *p*, and open *n* to pass the gas into *a*. When in *a*, fill *k* with Potash Solution, and allow it to run into *a* to absorb the Carbon Dioxide (CO_2) formed by the explosion. Pass the gas into *e* and take the reading. Calculate the volumes of Hydrogen and Marsh Gas by the following formulas:

Let C be the contraction of volume after the explosion.
Let D be the CO_2 formed by the explosion.

Then: $\frac{2\ C-4\ D}{3}$ = Hydrogen (H).

D = Marsh Gas (CH_4).

These calculations will give the volumes of H and CH_4 in the 20 Cc. taken for explosion. In order to calculate the H and CH_4 in the total volume of gas, multiply the above volumes by the residue left after absorbing the CO_2, O, and CO, and divide this result by the volume taken for explosion. After obtaining the percentages of CO_2, O, CO, H, and CH_4 subtract their sum from 100 to get the percentage of Nitrogen (N).

Example of Producer Gas Analysis.

CO_2 found by absorption with Potash,	4.8 Cc.
O found by absorption with Pyrogall,	.4 Cc.
CO found by absorption with Cuprous Chloride,	18.6 Cc.
	23.8 Cc.

100—23.8 = 76.2 Cc. residual gas.

Taken for explosion,	20.1 Cc.
Volume after adding Oxygen,	60. Cc.
Volume after explosion,	55.1 Cc.
Contraction,	4.9 Cc.
Volume after explosion,	55.1 Cc.
Volume after absorption with Potash,	54.8 Cc.
CO_2 formed by explosion,	.3 Cc.

Calculation $\frac{2\ C-4\ D}{3}$ = H in 20.1 Cc. taken for explosion.

D = CH_4 in 20.1 Cc. taken for explosion.

C = 4.9 Cc.

D = .3 Cc.

$$\frac{(2\times 4.9)-(4\times .3)}{3} = \frac{9.8-1.2}{3} = \frac{8.6}{3} = 2.9 \text{ H in } 20.1 \text{ Cc.}$$

Residual Gas $\frac{76.2\times 2.9}{20.1}$ = 10.9 Cc. H in 100 Cc. Gas.

$\frac{76.2\times .3}{20.1}$ = 1.1 Cc. CH_4 in 100 Cc. Gas.

Summary.

Carbon Dioxide, CO_2,	4.8
Oxygen, O,	.4
Carbonic Oxide, CO,	18.6
Hydrogen, H,	10.9
Marsh Gas, CH_4,	1.1
Nitrogen, N, by difference,	64.2
	100 per cent.

Conclusions.

In producer gas formed by forcing air or steam, or both through burning coal, the valuable constituents are Carbonic Oxide, Hydrogen, and Marsh Gas; the others are of no value and the producer should be managed so as to keep their percentages as low as possible. Over 5 per cent. of Carbon Dioxide, and over 0.5 per cent. of Oxygen should be regarded with suspicion as indicating in the former case a producer containing too little coal, and in the latter case the coal loosely packed, or leaks at the grate, allowing the air a too free passage.

WATER FOR USE IN STEAM BOILERS.

Waters which contain large proportions of the Carbonates and Sulphates of Lime and Magnesia are injurious to boilers, depositing a hard scale, which besides being a poor conductor of heat and thus causing waste of fuel, is the cause of the burning out of the fire tubes. When these salts are present in the water in considerable quantities, it is called a "Hard Water." In an analysis of boiler water, the following are determined: Hardness, Suspended Matter, Total Solids in solution, Chlorine in Chlorides, Sulphates, Silica, Iron Oxide and Alumina, Lime and Magnesia.

Sampling.—Allow the water to run from the hydrant or pump for about 10 minutes. Wash out a gallon bottle with the running water. Fill the bottle, and empty it three times. Then fill it again, and close it tightly with a cork.

Determination of Hardness.—The solutions required are a Standard Hard Water, and a Standard Soap Solution. Dissolve 1 gram of c. p. Calcium Carbonate in dilute Hydrochloric Acid, and evaporate to dryness on the water-bath, remove to a sand-bath and heat until all odor of acid has disappeared. Dissolve in a little

distilled water, wash the solution into a 1-liter graduated flask, fill to the mark with distilled water and mix well by shaking. Pour this solution into a clean, dry bottle and label it as follows:

STANDARD HARD WATER

1 Cc. = .001 Gm. $CaCO_3$.

Dissolve 10 grams of White Castile Soap in 90 per cent. alcohol, filter into a 1-liter graduated flask, and fill to the mark with more alcohol. Keep in a clean dry bottle, labeled:

STRONG SOAP SOLUTION.

To standardize the Soap Solution measure out 10 Cc. of the Standard Hard Water with a pipette, run it into a 6-ounce glass-stoppered bottle and add 90 Cc. distilled water. Drop the Soap Solution into this bottle from a burette, closing the bottle, and shaking it after each addition of the Soap Solution. Continue this until a permanent lather is formed, which will remain unbroken for at least five minutes. This lather must lightly cover the surface, and not be thick or frothy. From the number of Cc. and tenths of the Soap Solution used, calculate its value in terms of Carbonate of Lime. Suppose we found it took 19.5 Cc. of the Soap Solution to form the lather, we have: 19.5 Cc. Soap Solution equals 10 Cc. of the Hard Water or .01 gram $CaCO_3$. Therefore 1 Cc. equals .0005128 Gm. $CaCO_3$.

Take 100 Cc. of the water to be tested, and run it into a similar glass-stoppered bottle, drop in the Soap Solution exactly as described above. Multiply the volume used by the value of 1 Cc. The result will be the grams of $CaCO_3$ in the 100 Cc. of water. This result multiplied by 10000 will give the degrees of Total Hardness in parts per million.

Example:

1 Cc. Soap Solution found equal to .0005128 gram $CaCO_3$.

100 Cc. of Water required 7.5 Cc. Soap Solution to form lather.

$$7.5 \times .0005128 = .003846 \text{ gram } CaCO_3.$$

$$.003846 \times 10000 = 38.46 \text{ parts per million of } CaCO_3.$$

Water may be "hard" by reason of dissolved Carbon Dioxide, which can be removed by boiling. This is called "Temporary Hardness." To determine this, boil 100 Cc. of the water in a flask for one hour. Keep up the volume by adding from time to time boiled distilled water. Place the flask under a hydrant, and

allow cold water to run on the outside. When cool pour into the stoppered bottle and drop in the Soap Solution as above described. The number of grams of $CaCO_3$ found will be the Permanent Hardness, this subtracted from the Total Hardness gives the Temporary Hardness.

Suspended Matter.—Shake up the sample of water and if much suspended matter is seen, measure out 500 Cc. This is passed through a filter-paper which has been first dried in the oven at about 80° C. and then weighed. When the water has all passed through, wash three times with distilled water, remove again to the oven, and when dry weigh the paper. The increase of weight will be Suspended Matter, which multiplied by 2000 gives the parts per million in the water.

Total Solids in Solution.—While filtering the water as in the last paragraph, allow the filtrate from the Suspended Matter to run into a weighed platinum or porcelain dish. When the dish is nearly full remove it to a water-bath, and place a large beaker beneath the funnel. Evaporate the contents of the dish, and from time to time add some of the water from the beaker. When the whole has evaporated to dryness remove the dish to an air-bath and allow it to remain for one hour at a temperature of 130° C. Remove to a desiccator, and weigh when cool. Deduct the weight of the dish. The result will be the Total Solids in Solution. Multiply this by 2000 to get the result in parts per million.

Chlorine in Chlorides.—Measure 500 Cc. of the water into a porcelain dish, add a drop of Potassium Chromate Indicator, and from a burette drop in sufficient Standard Silver Nitrate to give the proper red color. (See Salt in Soda Ash, page 55). The number of Cc. and tenths of the Standard Solution used multiplied by .000606 gives the weight of Chlorine, this result multiplied by 2000 gives the Chlorine in parts per million.

Example, 500 Cc. of the water taken:

Standard Silver Nitrate 5.3 Cc. $\times$ .000606 = .0032118 Gm. Cl.

.0032118 $\times$ 2000 = 6.42 parts Cl per million.

Sulphates.—Measure 500 Cc. of the water into a porcelain dish, add 1 Cc. of dilute Hydrochloric Acid, and evaporate on the water-bath until the volume is reduced to about 200 Cc. Pour into a 250 Cc. beaker, and wash the dish into the beaker with a jet of hot water. Heat the contents of the beaker to boiling and

add 1 Cc. of hot solution of Barium Chloride. Allow the beaker to stand over night and filter. Proceed as described in Sodium Sulphate on page 56. The weight of the Barium Sulphate found, multiplied by 80 and divided by 232 gives the weight of Sulphates (SO_3) in the water, and this result multiplied by 2000 gives the parts per million.

Example, 500 Cc. of water taken:

Weight of crucible, cover and $BaSO_4$,	6.9615
Weight of crucible and cover,	6.9535
Weight of $BaSO_4$,	.0080

$$.0080 \times \tfrac{80}{232} = .002759.$$

$$.002759 \times 2000 = 5.518 \text{ parts } SO_3 \text{ per million.}$$

Silica, Alumina and Iron Oxide, Lime and Magnesia.—Measure 1000 Cc. of the water, and filter it into a porcelain dish, add 1 Cc. of concentrated Hydrochloric Acid, and evaporate to dryness on the water-bath. Dissolve the residue in 5 Cc. of dilute Hydrochloric Acid, and evaporate again to dryness. Redissolve in 5 Cc. dilute Hydrochloric Acid, and add 10 Cc. hot distilled water, filter through a 7 Cm. ashless filter-paper into a 50 Cc. beaker. Wash three times with a jet of hot water, and test the last drops of the filtrate with Silver Nitrate Solution. Ignite the filter in a weighed platinum crucible, cool in a desiccator, weigh and deduct the weight of the crucible, the result will be Silica (SiO_2) which multiplied by 1000 gives the Silica in parts per million.

Add a drop of concentrated Nitric Acid to the filtrate, boil and add enough strong Ammonia to give a slight odor, boil and filter through a 7 Cm. ashless filter-paper into a 100 Cc. beaker. Wash three times with hot water, or until the last drops of the filtrate give no reaction with Silver Nitrate Solution. Ignite the filter in a weighed porcelain crucible, cool and weigh. Deduct the weight of the crucible. The result is Alumina and Iron Oxide ($Al_2O_3 + Fe_2O_3$). Multiply this weight by 1000 to get the result in parts per million.

To the filtrate from the above add a few drops of strong Ammonia, boil, and while boiling add about 1 Cc. of Ammonium Oxalate Solution. Boil for a few minutes with constant stirring, and set aside for an hour. Filter through a 7 Cm. filter-paper, and wash with hot water until the last drops give no reaction with

Silver Nitrate Solution. Set the filtrate over a burner and allow it to evaporate. Place a 100 Cc. flask under the funnel and fill the funnel with hot dilute Sulphuric Acid (1 of acid, 5 of water). Fill the funnel three times with hot water. Place the flask under a burette, and titrate with weak Standard Permanganate. The number of Cc. and tenths of the Standard Solution used multiplied by .0005, will give the Lime (CaO), this result multiplied by 1000 gives the parts per million of Lime.

When the filtrate from the Calcium Oxalate precipitation has been reduced in volume to about 50 Cc. allow it to cool, add enough strong Ammonia to give a decided odor, then add 1 Cc. of Sodium Phosphate Solution, stir well for a short time, and allow to stand over night. Filter on a 7 Cm. ashless filter-paper, and proceed as directed on page 71 (Lime Analysis). The weight of the $Mg_2P_2O_7$ found, multiplied by 80 and divided by 222, gives the weight of Magnesia (MgO), and this result multiplied by 1000 gives the parts MgO per million.

Example, 1000 Cc. of water taken:

Residue insoluble after evaporation with HCl.

Weight of crucible and Silica,	7.1976
Weight of crucible,	7.1805
Weight of SiO_2,	.0171

$.0171 \times 1000 = 17.1$ parts SiO_2 per million.

Filtrate from Silica:

Weight of crucible and contents,	6.8132
Weight of crucible,	6.8091
Weight of $Al_2O_3 + Fe_2O_3$,	.0041

$.0041 \times 1000 = 4.1$ parts $Al_2O_3 + Fe_2O_3$ per million.

Filtrate from precipitation of Alumina and Iron Hydroxides:

Weak Standard Permanganate 12.1 Cc.

$12.1 \times .0005 = .00605$ CaO.

$.00605 \times 1000 = 6.05$ parts CaO per million.

Filtrate from Calcium Oxalate precipitation:

Weight of crucible and contents,	14.8147
Weight of crucible,	14.8040
Weight of $Mg_2P_2O_7$,	.0107

$.0107 \times \frac{80}{222} = .00386$ MgO.

$.00386 \times 1000 = 3.86$ parts MgO per million.

Calculations.

It is customary to report the impurities of water in terms of grains per U. S. gallon of 231 cubic inches. The number of grains in such a gallon has been fixed upon as 58318. The impurities have been determined in parts per million, or in milligrams per liter since 1 milligram is one-millionth of a liter. Therefore to convert into grains per gallon multiply by 58318 and divide by one million. Taking the results found above we have:

	Parts per Million.				Grains per Gallon.
Total Hardness,	38.46	×	$\frac{58318}{1000000}$	=	2.2
Chlorine in Chlorides,	6.42	×	"	=	.37
Sulphates,	5.518	×	"	=	.32
Silica,	17.1	×	"	=	1.
Alumina and Iron Oxide,	4.1	×	"	=	.24
Lime,	6.05	×	"	=	.35
Magnesia,	3.86	×	"	=	.22

APPENDIX.

SPECIFIC GRAVITY.

The Specific Gravity or Density of a body is its weight compared with the weight of an equal volume of distilled water usually determined at 15° C. To determine the density of a solid not soluble in water proceed as follows. Hang the body by means of a fine thread or hair from the hook of the Balance beam (Fig. 85, *c*). Place sufficient weights on the opposite pan to balance, and note the weight of the suspended body. Then place the wooden support or bench *a* (Fig. 86) across the pan. On the

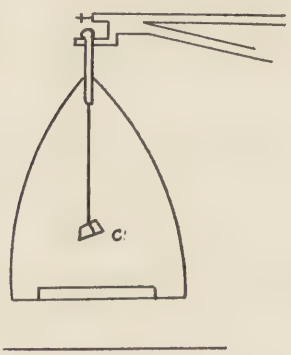


Fig. 85.

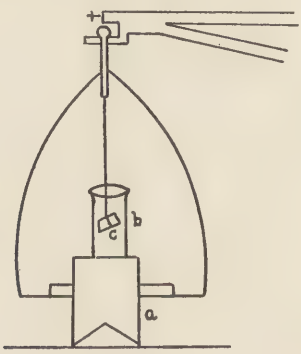


Fig. 86.

bench place the beaker *b* containing boiled distilled water in which the body *c* is entirely immersed, not allowing it to touch the bottom or sides of the beaker and removing all air bubbles. From the opposite pan remove sufficient weights to balance again. Observe the loss of weight. Divide the first weight by this loss, the result will be the Specific Gravity or Density of the body.

Example, Bottle Glass:

Weight in air,	1.6195
Weight in water,	.9776
Loss of weight,	.6419

1.6195 ÷ .6419 = 2.523 Specific Gravity, or in other words, this body is 2.523 times as heavy as an equal volume of water.

If the density of a liquid is desired, use is made of a carefully weighed and graduated bottle known as the Specific Gravity Bottle. Figure 87 shows two forms. The form *a* is provided with a glass-stopper pierced with a capillary opening. To use this form fill it with the liquid and insert the stopper, allowing the excess of liquid to escape through the capillary passage. Wipe

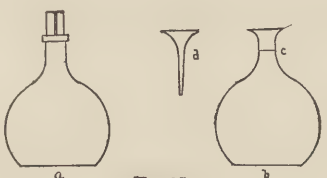


Fig. 87.

Fig. 88.

Fig. 89.

the outside dry and weigh the bottle. From this weight deduct the weight of the bottle. The result will be the weight of the volume of liquid which the bottle contains. Divide this weight by the weight of an equal volume of distilled water; the result will be the density of the liquid.

Example, Specific Gravity of a Sample of Oil:
Bottle contains 100 grams of distilled water.

Weight of bottle and oil,	110.5332
Weight of bottle,	20.3821
Weight of oil,	90.1511

90.1511 ÷ 100 = .901511 or density of the oil.

The form *b* (Fig. 87) is also used. It has no stopper, but a mark *c* is engraved on the neck indicating the point to which the bottle must be filled. The funnel *d* is used in filling. This form is capable of quite as accurate use as the other, and has the advantage that no liquid is spilled on the outside, which may cause trouble in some cases. Instead of weighing these bottles, a counterpoise may be provided.

The Hydrometer (Fig. 88) is also used to determine the density of liquids. It is a glass instrument having a bulb *b* filled with mercury or shot, a wide part *c*, and a stem *d* which contains a paper scale divided into spaces representing specific gravity degrees and fractions. When the hydrometer is placed in the liquid to be tested it sinks until the level of the liquid coincides with the mark on the scale corresponding to the proper specific gravity. The Scales of Hydrometers are graduated differently. Some show direct specific gravity, others are marked in degrees of Beaumé, Twaddel, Brix, etc., according to the purpose desired.

Figure 89 shows a convenient jar for use with the hydrometer. It is long and narrow in shape, and requires the least possible volume of the liquid.

If the density of a body soluble in water is desired, we make use of a liquid in which the body is insoluble. Finely pulverize the body, and place it in the bottle *a*. Insert the stopper and weigh. Deduct the weight of the bottle; the remainder will be the weight of the body. Fill the bottle with the suitable liquid, the density of which is known, shake to expel air, insert the stopper and weigh again. Deduct the weight of the body; the remainder will be the weight of the liquid in the bottle. Subtract this from the weight of liquid which would be found if the bottle were entirely full of liquid. The result will be the liquid displaced by the body. Multiply the density of the liquid by the weight of the body, and divide the result by the weight of the liquid displaced; the result will be the density of the body.

Example, Soda Ash 58 per cent. taken for the determination:
Liquid used, Naptha with a density of .6935.

Weight of bottle and Ash,	25.9821
Weight of bottle,	20.3821
Weight of Ash,	5.6000 grams.

Weight of bottle, Ash and Naptha,	94.0141
Weight of bottle and Ash,	25.9821
Weight of Naptha in bottle,	68.0320

Density of Naptha .6935.

Capacity of bottle 100 Cc., hence it can hold 69.35 grams of Naptha.

Weight of 100 Cc. Naptha,	69.3500
Weight of Naptha in bottle,	68.0320
Weight of Naptha, displaced,	1.3180

$(5.6 \times .6935) \div 1.318 = 2.94$, Density of Ash.

TABLES.

INTERNATIONAL ATOMIC WEIGHTS.

REPORTED BY THE INTERNATIONAL COMMITTEE.

Proceedings of the London Chemical Society, January 29, 1903, p. 4.

Name.	O=16.	H=1.	Name.	O=16.	H=1.
Aluminium	27.1	26.9	Molybdenum	96.0	95.3
Antimony	120.2	119.3	Neodymium	143.6	142.5
Argon	39.9	39.6	Neon	20.	19.9
Arsenic	75.0	74.4	Nickel	58.7	58.3
Barium	137.4	136.4	Nitrogen	14.04	13.93
Bismuth	208.5	206.9	Osmium	191.	189.6
Boron	11.	10.9	Oxygen	16.00	15.88
Bromine	79.96	79.36	Palladium	106.5	105.7
Cadmium	112.4	111.6	Phosphorus	31.0	30.77
Cæsium	133.	132.	Platinum	194.8	193.3
Calcium	40.1	39.8	Potassium	39.15	38.86
Carbon	12.00	11.91	Praseodymium	140.5	139.4
Cerium	140.	139.	Radium	225.	223.3
Chlorine	35.45	35.18	Rhodium	103.0	102.2
Chromium	52 1	51.7	Rubidium	85.4	84.8
Cobalt	59.0	58.56	Ruthenium	101.7	100.9
Columbium (Niobium)	94.	93.3	Samarium	150.	148.9
Copper	63.6	63.1	Scandium	44.1	43.8
Erbium	166.	164.8	Selenium	79.2	78.6
Fluorine	19.	18.9	Silicon	28.4	28.2
Gadolinium	156.	155.	Silver	107.93	107.12
Gallium	70.	69.5	Sodium	23.05	22.88
Germanium	72.5	71.9	Strontium	87.6	86.94
Glucinum (Beryllium)	9.1	9.03	Sulphur	32.06	31.83
Gold	197.2	195.7	Tantalum	183.	181.6
Helium	4.	4.	Tellurium	127.6	126 6
Hydrogen	1.008	1.000	Terbium	160.	158.8
Indium	114.	113.1	Thallium	204.1	202.6
Iodine	126.85	125.90	Thorium	232.5	230.8
Iridium	193.0	191.5	Thulium	171.	169.7
Iron	55.9	55.5	Tin	119.0	118.1
Krypton	81.8	81.2	Titanium	48.1	47.7
Lanthanum	138.9	137.9	Tungsten	184.0	182.6
Lead	206.9	205.35	Uranium	238.5	236.7
Lithium	7.03	6.98	Vanadium	51.2	50 8
Magnesium	24.36	24.18	Xenon	128.	127.
Manganese	55.0	54.6	Ytterbium	173.0	171.7
Mercury	200.0	198.5	Yttrium	89.0	88.3
			Zinc	65.4	64.9
			Zirconium	90.6	89.9

THERMOMETER COMPARISONS.

To convert Centigrade degrees to Fahrenheit degrees, multiply by 9, divide by 5, and add 32 to the result.

To convert Fahrenheit degrees to Centigrade degrees, subtract 32, then multiply the result by 5, and divide by 9.

°C	°F	°C	°F	°C	°F	°C	°F
500	932	74	165.2	44	111.3	14	57.2
400	752	73	163.4	43	109.4	13	55.4
300	572	72	161.6	42	107.6	12	53.6
200	392	71	159.8	41	105.8	11	51.8
100	212	70	158	40	104	10	50
99	210.2	69	156.2	39	102.2	9	48.2
98	208.4	68	154.4	38	100.4	8	46.4
97	206.6	67	152.6	37	98.6	7	44.6
96	204.8	66	150.8	36	96.8	6	42.8
95	203	65	149	35	95	5	41
94	201.2	64	147.2	34	93.2	4	39.2
93	199.4	63	145.4	33	91.4	3	37.4
92	197.6	62	143.6	32	89.6	2	35.6
91	195.8	61	141.8	31	87.8	1	33.8
90	194	60	140	30	86	0	32
89	192.2	59	138.2	29	84.2	—1	30.2
88	190.4	58	136.4	28	82.4	—2	28.4
87	188.6	57	134.6	27	80.6	—3	26.6
86	186.8	56	132.8	26	78.8	—4	24.8
85	185	55	131	25	77	—5	23
84	183.2	54	129.2	24	75.2	—6	21.2
83	181.4	53	127.4	23	73.4	—7	19.4
82	179.6	52	125.6	22	71.6	—8	17.6
81	177.8	51	123.8	21	69.8	—9	15.8
80	176	50	122	20	68	—10	14
79	174.2	49	120.2	19	66.2	—11	12.2
78	172.4	48	118.4	18	64.4	—12	10.4
77	170.6	47	116.6	17	62.6	—13	8.6
76	168.8	46	114.8	16	60.8	—14	6.8
75	167	45	113	15	59	—15	5

BAUMÉ'S HYDROMETER SCALES

WITH CORRESPONDING SPECIFIC GRAVITIES

For Liquids Heavier than Water. Temp. 15° C.				For Liquids Lighter than Water. Temp. 15° C.			
Baumé.	Specific Gravity.	Baumé.	Specific Gravity.	Baumé.	Specific Gravity.	Baumé.	Specific Gravity.
0	1.0000	38	1.3551	10	1.0000	48	.7865
1	1.0069	39	1.3679	11	.9929	49	.7821
2	1.0140	40	1.3810	12	.9859	50	.7777
3	1.0211	41	1.3942	13	.9790	51	.7735
4	1.0284	42	1.4078	14	.9722	52	.7692
5	1.0357	43	1.4216	15	.9655	53	.7650
6	1.0432	44	1.4356	16	.9589	54	.7609
7	1.0507	45	1.4500	17	.9524	55	.7568
8	1.0584	46	1.4646	18	.9459	56	.7527
9	1.0662	47	1.4796	19	.9396	57	.7487
10	1.0741	48	1.4948	20	.9333	58	.7447
11	1.0821	49	1 5104	21	.9272	59	.7407
12	1.0902	50	1.5263	22	.9211	60	.7368
13	1.0985	51	1.5426	23	.9150	61	.7329
14	1.1069	52	1.5591	24	.9091	62	.7292
15	1.1154	53	1.5761	25	.9032	63	.7254
16	1.1240	54	1.5934	26	.8974	64	.7217
17	1.1328	55	1.6111	27	.8917	65	.7179
18	1.1417	56	1.6292	28	.8861	66	.7143
19	1.1508	57	1.6477	29	.8805	67	.7107
20	1.1600	58	1.6667	30	.8750	68	.7071
21	1.1694	59	1.6860	31	.8696	69	.7035
22	1.1789	60	1.7059	32	.8642	70	.7000
23	1.1885	61	1.7262	33	.8589	71	.6965
24	1.1983	62	1.7470	34	.8537	72	.6931
25	1.2083	63	1.7683	35	.8485	73	.6897
26	1.2185	64	1.7901	36	.8433	74	.6863
27	1.2288	65	1.8125	37	.8383	75	.6829
28	1.2393	66	1.8354	38	.8333	76	.6796
29	1.2500	67	1.8590	39	.8285	77	.6763
30	1.2609	68	1.8831	40	.8234	78	.6730
31	1.2719	69	1.9079	41	.8187	79	.6698
32	1.2832	70	1.9333	42	.8139	80	.6666
33	1.2946	71	1.9595	43	.8092	.....	.....
34	1.3063	72	1.9863	44	.8046	.....	.....
35	1.3182	73	2.0139	45	.8000	.....	.....
36	1.3303	74	2.0423	46	.7955	.....	.....
37	1.3426	75	2.0714	47	.7909	.....	.....

PERCENTAGE AND GRAVITY OF SULPHURIC ACID.

Table showing percentages of Actual Sulphuric Acid (H_2SO_4) corresponding to various Specific Gravities of Aqueous Sulphuric Acid (Bineau); Otto. Temp. 15°C.

Specific Gravity.	Per Cent.	Specific Gravity.	Per Cent.	Specific Gravity.	Per Cent.	Specific Gravity.	Per Cent.
1.8426	100	1.675	75	1.398	50	1.182	25
1.842	99	1.663	74	1.3886	49	1.174	24
1.8406	98	1.651	73	1.379	48	1.167	23
1.840	97	1.639	72	1.370	47	1.159	22
1.8384	96	1.627	71	1.361	46	1.1516	21
1.8376	95	1.615	70	1.351	45	1.144	20
1.8356	94	1.604	69	1.342	44	1.136	19
1.834	93	1.592	68	1.333	43	1.129	18
1.831	92	1.580	67	1.324	42	1.121	17
1.827	91	1.568	66	1.315	41	1.1136	16
1.822	90	1.557	65	1.306	40	1.106	15
1.816	89	1.545	64	1.2976	39	1.098	14
1.809	88	1.534	63	1.289	38	1.091	13
1.802	87	1.523	62	1.281	37	1.083	12
1.794	86	1.512	61	1.272	36	1.0756	11
1.786	85	1.501	60	1.264	35	1.068	10
1.777	84	1.490	59	1.256	34	1.061	9
1.767	83	1.480	58	1.2476	33	1.0536	8
1.756	82	1.469	57	1.239	32	1.0464	7
1.745	81	1.4586	56	1.231	31	1.039	6
1.734	80	1.448	55	1.223	30	1.032	5
1.722	79	1.438	54	1.215	29	1.0256	4
1.710	78	1.428	53	1.2066	28	1.019	3
1.698	77	1.418	52	1.198	27	1.013	2
1.686	76	1.408	51	1.190	26	1.0064	1

PERCENTAGES AND GRAVITY OF HYDROCHLORIC ACID.

Table showing the percentages of Hydrochloric Acid in Aqueous Solutions of the Gas of Various Specific Gravities. Ure; Temp. 15° C.

Specific Gravity.	HCl Per Cent.	Specific Gravity.	HCl Per Cent.	Specific Gravity.	HCl Per Cent.	Specific Gravity.	HCl Per Cent.
1.200	40.777	1.1515	30.582	1.1000	20.388	1.0497	10.194
1.1982	40.369	1.1494	30.174	1.0980	19.980	1.0477	9.786
1.1964	39.961	1.1473	29.767	1.0960	19.572	1.0457	9.379
1.1946	39.554	1.1452	29.359	1.0939	19.165	1.0437	8.971
1.1928	39.146	1.1431	28.951	1.0919	18.757	1.0417	8.563
1.1910	38.738	1.141	28.544	1.0899	18.349	1.0397	8.155
1.1893	38.330	1.1389	28.136	1.0879	17.941	1.0377	7.747
1.1875	37.923	1.1369	27.728	1.0859	17.534	1.0357	7.340
1.1857	37.516	1.1349	27.341	1.0838	17.126	1.0337	6.932
1.1846	37.108	1.1328	26.913	1.0818	16.718	1.0318	6.524
1.1822	36.700	1.1308	26.505	1.0798	16.310	1.0298	6.116
1.1802	36.292	1.1287	26.098	1.0778	15.902	1.0279	5.709
1.1782	35.884	1.1267	25.690	1.0758	15.494	1.0259	5.301
1.1762	35.476	1.1247	25.282	1.0738	15.087	1.0239	4.893
1.1741	35.068	1.1226	24.874	1.0718	14.679	1.0220	4.486
1.1721	34.660	1.1206	24.466	1.0697	14.271	1.0200	4.078
1.1701	34.252	1.1185	24.058	1.0677	13.863	1.0180	3.670
1.1681	33.845	1.1164	23.650	1.0657	13.456	1.0160	3.262
1.1661	33.438	1.1143	23.242	1.0637	13.049	1.0140	2.854
1.1641	33.029	1.1123	22.834	1.0617	12.641	1.0120	2.447
1.1620	32.621	1.1102	22.426	1.0597	12.233	1.0100	2.039
1.1599	32.213	1.1082	22.019	1.0577	11.825	1.0080	1.631
1.1578	31.805	1.1061	21.611	1.0557	11.418	1.0060	1.124
1.1557	31.398	1.1041	21.203	1.0537	11.010	1.0040	.816
1.1536	30.990	1.1020	20.796	1.0517	10.602	1.0020	.408

PERCENTAGES AND GRAVITY OF NITRIC ACID.

Table showing the percentages of Nitric Acid (HNO_3) in Aqueous Solutions of Various Specific Gravities. Kolb, Ann. Ch. Phys. (4), 136. Temp. 15° C.

HNO_3 Per Cent.	Specific Gravity.	HNO_3 Per Cent.	Specific Gravity.	HNO_3 Per Cent.	Specific Gravity.	HNO_3 Per Cent.	Specific Gravity.
100.00	1.530	80.96	1.463	59.59	1.372	39.00	1.244
99.84	1.530	80.00	1.460	58.88	1.368	37.95	1.237
99.72	1.530	79.00	1.456	58.00	1.363	36.00	1.225
99.52	1.529	77.60	1.451	57.00	1.358	35.00	1.218
97.89	1.523	76.00	1.445	56.10	1.353	33.86	1.211
97.00	1.520	75.00	1.442	55.00	1.346	32.00	1.198
96.00	1.516	74.01	1.438	54.00	1.341	31.00	1.192
95.27	1.514	73.00	1.435	53.81	1.339	30.00	1.185
94.00	1.509	72.39	1.432	53.00	1.335	29.00	1.179
93.01	1.506	71.24	1.429	52.33	1.331	28.00	1.172
92.00	1.503	69.96	1.423	50.99	1.323	27.00	1.166
91.00	1.499	69.20	1.419	49.97	1.317	25.71	1.157
90.00	1.495	68.00	1.414	49.00	1.312	23.00	1.138
89.56	1.494	67.00	1.410	48.00	1.304	20.00	1.120
88.00	1.488	66.00	1.405	47.18	1.298	17.47	1.105
87.45	1.486	65.07	1.400	46.64	1.295	15.00	1.089
86.17	1.482	64.00	1.395	45.00	1.284	13.00	1.077
85.00	1.478	63.59	1.393	43.53	1.274	11.41	1.067
84.00	1.474	62.00	1.386	42.00	1.264	7.22	1.045
83.00	1.470	61.21	1.381	41.00	1.257	4.00	1.022
82.00	1.467	60.00	1.374	40.00	1.251	2.00	1.010

COMPARISON OF WEIGHTS AND MEASURES, U. S. PHARMACOPŒIA.

Measures of Length.

1 Meter = 39.370432 inches.
1 Decimeter = 3.937043 "
1 Centimeter = 0.393704 "
1 Millimeter = 0.039370 "
1 Yard (or 36 inches) = 0.91439 Meter.
1 Foot (or 12 inches) = 30.48 Centimeters.

Inches.		*Centimeters.*	*Inches.*		*Centimeters.*	*Inches.*		*Millimeters.*
11	=	27.9	5	=	12.7	$\frac{1}{2}$	=	12.5
10	=	25.4	4	=	10.2	$\frac{1}{4}$	=	6.25
9	=	22.9	3	=	7.6	$\frac{1}{8}$	=	3.12
8	=	20.3	2	=	5.1	$\frac{1}{16}$	=	1.54
7	=	17.8	1	=	2.5	$\frac{1}{25}$	=	1.00
6	=	15.2						

Measures of Capacity.

Cubic Centimeters.		*Fluid Ounces.*	*Cubic Centimeters.*		*Fluid Drachms.*	*Cubic Centimeters.*		*Minims.*
1000	=	33.81	25	=	6.76	3	=	48.69
900	=	30.43	20	=	5.41	2	=	32.46
800	=	27.05	15	=	4.06	1	=	16.23
700	=	23.67	10	=	2.71	0.75	=	12.17
600	=	20.29	9	=	2.43	0.50	=	8.12
500	=	16.90	8	=	2.16	0.25	=	4.06
400	=	13.53	7	=	1.89	0.20	=	3.25
300	=	10.11	6	=	1.62	0.15	=	2.43
200	=	6.76	5	=	1.35	0.10	=	1.62
100	=	3.38	4	=	1.08	0.05	=	0.81
50	=	1.69						
10	=	.34						
1	=	.03						

Minims.		*Cubic Centimeters.*	*Minims.*		*Cubic Centimeters.*	*Minims.*		*Cubic Centimeters.*
1	=	0.06	8	=	0.49	35	=	2.16
2	=	0.12	9	=	0.55	40	=	2.46
3	=	0.18	10	=	0.62	45	=	2.77
4	=	0.25	15	=	0.92	50	=	3.08
5	=	0 31	20	=	1.23	55	=	3.39
6	=	0.37	25	=	1.54	60	=	3.70
7	=	0.43	30	=	1.85			

Fluid Drachms.		*Cubic Centimeters.*	*Fluid Ounces.*		*Cubic Centimeters.*	*Fluid Ounces.*		*Cubic Centimeters.*
1	=	3.70	1	=	29.57	9	=	266.10
2	=	7.39	2	=	59.10	10	=	295.68
3	=	11.09	3	=	88.67	12	=	354.82
4	=	14.79	4	=	118.24	16	=	473 11
5	=	18.48	5	=	147.81	24	=	709.80
6	=	22.18	6	=	177.39	32	=	946.38
7	=	25.88	7	=	206.96	64	=	1892.75
8	=	29.57	8	=	236.53	128	=	3785.51

Measures of Weight.

Grams.	*Troy Grains.*	*Grams.*	*Troy Grains.*	*Grams.*	*Troy Grains.*	*Grams.*	*Troy Grains.*
0.0010	= 0.015	0.0100	= 0.154	0.10	= 1.543	1	= 15.432
0.0013	= 0.019	0.0125	= 0.193	0.12	= 1.852	2	= 30.865
0.0015	= 0.023	0.0150	= 0.231	0.15	= 2.315	3	= 46.297
0.0020	= 0.031	0.0200	= 0.309	0.20	= 3.086	4	= 61.729
0.0025	= 0.039	0.0250	= 0.386	0.25	= 3.858	5	= 77.162
0.0030	= 0.046	0.0300	= 0.463	0.30	= 4.630	6	= 92.594
0.0035	= 0.054	0.0350	= 0.540	0.35	= 5.401	7	= 108.026
0.0040	= 0.062	0.0400	= 0.617	0.40	= 6.173	8	= 123.459
0.0045	= 0.069	0.0450	= 0.694	0.50	= 7.716	9	= 138.891
0.0050	= 0.077	0.0500	= 0.772	0.60	= 9.259	10	= 154.323
0.0055	= 0.085	0.0550	= 0.849	0.70	= 10.803	20	= 308.647
0.0060	= 0.093	0.0600	= 0.926	0.80	= 12.346	30	= 462.970
0.0065	= 0.100	0.0650	= 1.003	0.90	= 13.899	40	= 617.294
0.0070	= 0.108	0.0700	= 1.080			50	= 771.617
0.0075	= 0.116	0.0750	= 1.157			100	= 1543.235
0.0080	= 0.123	0.0800	= 1.235			200	= 3086.470
0.0085	= 0.131	0.0850	= 1.312			500	= 7716.174
0.0090	= 0.139	0.0900	= 1.389			750	= 11574.262
0.0095	= 0.147	0.0950	= 1.466			1000	= 15432.350

Troy Grains.		*Grams.*
$\frac{1}{64}$	=	0.00101
$\frac{1}{60}$	=	0.00108
$\frac{1}{50}$	=	0.00130
$\frac{1}{40}$	=	0.00162
$\frac{1}{32}$	=	0.00202
$\frac{1}{30}$	=	0.00216
$\frac{1}{24}$	=	0.00270
$\frac{1}{20}$	=	0.00324
$\frac{1}{16}$	=	0.00405
$\frac{1}{12}$	=	0.00540
$\frac{1}{10}$	=	0.00648

Troy Grains.		*Grams.*
$\frac{1}{8}$	=	0.00810
$\frac{1}{5}$	=	0.01296
$\frac{1}{4}$	=	0.01620
$\frac{2}{5}$	=	0.03240
$\frac{3}{4}$	=	0.04860
1	=	0.06480
2	=	0.12960
3	=	0.19440
4	=	0.25920
5	=	0.32399
6	=	0.38879

Troy Grains.		*Grams.*
7	=	0.45359
8	=	0.51839
9	=	0.58319
10	=	0.64799
20	=	1.296
30	=	1.944
40	=	2.592
50	=	3.240

Drachms.		*Grams.*
1	=	3.888
2	=	7.776
3	=	11.664
4	=	15.552
5	=	19.440
6	=	23.328
7	=	27.216

Ounces.		*Grams.*
1	=	31.103
2	=	62.207
3	=	93.310
4	=	124.414
5	=	155.517

Ounces.		*Grams.*
6	=	186.621
7	=	217.724
8	=	248.823
9	=	279.931
10	=	311.035

Grams.		*Avoirdupois. Ounces.*	*Grains.*
28.35	=	1	
29	=	1	10
30	=	1	25½
35	=	1	103
40	=	1	180
50	=	1	334
60	=	2	50½
70	=	2	205
80	=	2	359
90	=	3	76½
100	=	3	230½

Grams.		*Avoirdupois. Ounces.*	*Grains.*
200	=	7	24
250	=	8	358
300	=	10	255
350	=	12	151½
400	=	14	48
450	=	15	382
500	=	17	279
600	=	21	72
700	=	24	303
800	=	28	96
900	=	31	326½
1000	=	35	120

Avoirdupois. Ounces.		*Grams.*
$\frac{1}{16}$	=	1.772
$\frac{1}{8}$	=	3.544
$\frac{1}{4}$	=	7.088
$\frac{1}{2}$	=	14.175
1	=	28.350
2	=	56.699
3	=	85.049
4	=	113.398
5	=	141.748
6	=	170.098

Avoirdupois. Ounces.		*Grams.*
7	=	198.447
8	=	226.796
9	=	255.146
10	=	283.496
11	=	311.846
12	=	340.195
13	=	368.544
14	=	396.894
15	=	425.243
16	=	453.592

Avoirdupois. Grams.	*Pounds.*		*Grams.*
198.447	1	=	453.592
226.796	2	=	907.18
255.146	3	=	1360.78
283.496	4	=	1814.37
311.846	5	=	2267.96
340.195	6	=	2721.55
368.544	7	=	3175.14
396.894	8	=	3628.74
425.243	9	=	4082.33
453.592	10	=	4535.92

NOTES UPON THE USE AND CARE OF PLATINUM WARE.

By Baker & Co.,

Mfrs. of Hammered Platinum Ware, Newark, N. J.

It is important to remember that, although platinum is not oxidized in the air at any temperature, nor attacked by any single acid, yet there are many substances that attack and combine with it at comparatively low temperatures.

The caustic alkalies, the alkaline earths, nitrates and cyanides, and especially the hydrates of barium and lithium, attack platinum at a red heat, although the alkaline carbonates have no effect at the highest temperatures. Sulphur, in the absence of alkalies, has no action, but phosphorous and arsenic attack platinum when heated with it.

Direct contact of platinum with burning charcoal should be avoided, since the silicon reduced from the charcoal ash unites with platinum, making it brittle and liable to fracture.

Also contact with compounds of the easily reducible metals is especially dangerous at high temperatures, as alloys with platinum having a low fusing point are readily formed. This is especially true of lead.

Heating of platinum with spirit lamps is preferable to the use of ordinary gas. When gas is used, care should be taken to have the supply of air sufficient to insure complete combustion, since, with a flame containing free carbon, the platinum suffers deterioration by the formation of a carbide of platinum, which, oxidizing later, blisters the metal. For this reason, also, the inner cone or reducing flame should not be in contact with the metal.

The loosening effect of the Bunsen flame upon the surface of platinum exposed to its action produces the familiar gray appearance which cannot be removed except by burnishing. Platinum triangles often become gray and very brittle from the same cause. Systematic application of moist sand to all articles affected in this way, after use, will keep them in prime condition and materially prolong their life, with but a trifling loss in weight.

CLEANING PLATINUM WARE.

Every careful analyst of necessity uses clean utensils. A habit of cleaning and polishing platinum dishes immediately after using is easily formed, and repays the user with increased confidence in his work as well as in the prolonged life of the article.

Rubbing the surface of platinum with moist sea sand (round grains only), applied with the finger, serves to remove most impurities and to polish the metal without material loss.

Fusing bisulphate of potash or borax in the dish and then boiling in water and polishing as above with sand is recommended by Gmelin. When it is desired to clean the outer surface of dishes in this manner, they must be placed in dishes of sufficient size to allow the fused flux to completely envelope the article to be cleaned.

Sodium amalgam possesses the property of wetting platinum without amalgamating with it, even when other metals are purposely added to the amalgam. This substance is, therefore, useful for effecting a quick and thorough cleansing of platinum. The amalgam is gently rubbed upon the metal with a cloth and then moistened with water, which oxidizes the sodium and leaves the mercury free to alloy with foreign metals. The mercury is then wiped off and the dish cleaned and polished with sand, as above described.

If the existence of a base metal alloyed with the platinum is suspected, immerse the article in question first in boiling HCl for a few minutes, then, after thorough rinsing with clean water, immerse in boiling HNO_3 acid free from chlorine.

If the dish is unaffected in weight or appearance, and the acid baths fail to give reactions for the base metals, their absence in appreciable quantities is assured.

INDEX.

A

B

C

D

Q

R

S